

LONGHORN ARMY AMMUNITION PLANT

MARSHALL, TEXAS

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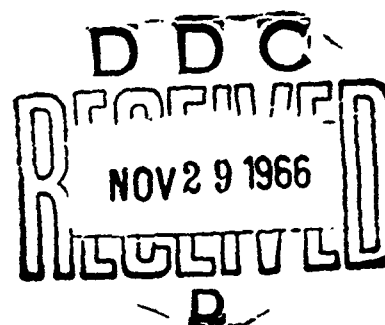
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HISTORICAL SUMMARY SAFETY DATA PERTINENT TO MANUFACTURE AND LOADING OF SOLID PROPELLANT

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May 1964



THIOKOL CHEMICAL CORPORATION

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Thiokol Chemical Corporation
Longhorn Division
Marshall, Texas

HISTORICAL SUMMARY
SAFETY DATA PERTINENT TO
MANUFACTURE AND LOADING
OF
SOLID PROPELLANT


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SAFETY SECTION

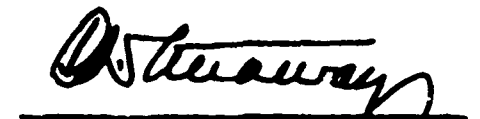
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INTRODUCTION

This document is a summary of incidents occurring in the manufacture and loading of solid propellant with the safety data developed from such incidents, as well as from tests and studies conducted to determine hazards.

References and sources are cited for more detailed information on the subjects covered and the verification of conclusions reached.

The publication of this document is not meant to imply that this installation recommends, endorses, or concurs in the findings, conclusions, or analyses obtained, but rather to accumulate the information reported as a historical reference source for any safety aspects developed by contributors.

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INCIDENTS

SPONTANEOUS IGNITION, EXCESSIVE HEAT, ETC.

- 10:10 (1) Contaminated Waste Barrel Fire: Thiokol, Huntsville Division, 1960.
A fire occurred in a contaminated waste barrel located outside the laboratory. The fire spread to four other drums containing contaminated waste. The fire evidently occurred from spontaneous combustion. No injuries. Probable Cause: Lithium aluminum hydride.
Annual Safety Report, 1960, Thiokol, Huntsville Division.
- 10:10 (2) Fire in Waste Container: Longhorn, 1959.
A fire originated one Sunday afternoon in a waste container located outside the Longhorn Chemistry Laboratory. The fire ignited some other containers of contaminated waste. The cause is believed to have been spontaneous combustion.
- 10:10 (3) Propellant Curing Oven Fire (samples): Thiokol, Wasatch Division, Sept. 1960.
Propellant samples (approximately 60 pounds) ignited in a curing oven. Probably caused by malfunction of heat controlling system.
- 10:10 (4) Cutting Propellant Samples with Hacksaw, Sample Ignited: Reaction Motors, Denville, N. J., 19 January 1960.
A small sample of compressed molded propellant (20% PIB, 58.9% HAZ, and 21.1% TANG materials) ignited when manually cutting a 1½" diameter x 3" long sample with a hacksaw. The laboratory technician received burns on his face and hands. For probable causes, see report.
Longhorn Safety Section File 341-1/304.4.6.
- 10:10 (5) Ignition of Waste Container on Building Pad: Thiokol, Wasatch Division.
Forklift operator placed a pallet of containers filled with propellant on an elevated explosive waste pad. When pulling the forks from the pallet, he must have scooted the pallet across the floor of the pad and ignited some propellant on the bottom of the pallet. He didn't notice the initial burning but after leaving he looked back and saw the containers burning.
- 10:10 (6) Ignition of Small Amount of Propellant on Floors, Equipment, by Friction or Pinching: Thiokol, Longhorn Division, 3 March 1964.
Building 45-E: When rolling a cart out of an oven bay, the casters rolled over a small amount of uncured solid propellant (polysulfide/AP) and ignited it. Building 54-F, 3-24-64: Employee walking in corridor in front of a cut-back bay ignited (popping noise) small particles of cured solid propellant (polysulfide/AP) by frictional heat between his shoe and concrete floor. Building 45-E, 4-9-64: Engineer stacking contaminated casting fixture parts ignited a small amount of uncured solid propellant (Perahine) by pinching between the metal parts. In all three cases the fire did not spread due to the small quantity of waste propellant permitted on the floor and equipment.

MIXER FIRES AND EXPLOSIONS

10:11 For causes, theory, etc., see 21:15

- 10:11 (1) Mixer Fire: Naval Propellant Plant, 1 September 1954.
200-gallon, J. H. Day mixer. Carlock #234 plain yarn packing rings, five in each gland. Propellant binder system; nitrocellulose colloids with an ether-diphenylamine mixture. While processing reworked cannon powder, fire was detected by employee who was in the process of discharging the mixer. The J. H. Day mixers installed in 1942 have blades keyed to the drive shaft from the inside of the mixer. The key had fallen out of the keyway, and it is believed the cause of the fire was the friction created by slippage of the drive shaft within the hub of the blade.
Reference 11, page 78.
- 10:11 (2) Mixer Fire: Hercules Powder Company, 31 January 1954.
Readco Tangential Sigma Blade mixer. Double-base propellant. Mixer No. 6 in Building 3508 fired while processing M-10 single-base propellant. The mixer was charged with remix and solvent (acetone-alcohol and ether) and had gone through the required soaking time. Several minutes after mixer was started, the fire occurred. No apparent cause could be found for this incident.
Reference 11, page 31.
- 10:11 (3) Mixer Explosion: Astrodyne, Inc., McGregor, Texas, 25 November 1955.
20-gallon Baker Perkins mixer. 100 pounds ammonium perchlorate propellant. Six injuries. \$223,500 damages. Possible causes: 1) propellant material striking ram or becoming pinched between ram or side of mixer bowl; 2) stationary friction ring in blade shaft packing glands may have become overheated, igniting propellant which had accumulated within the gland—one stationary friction ring had a shattered face and there was evidence of much heat in this area; 3) metal-to-metal contact of parts inside the mixer bowl; 4) impurities. Corrective Actions: Discontinue use of ram on sensitive propellants, set up cleaning schedule for packing glands, replace metal-to-metal seal in packing glands, etc.
Reference 12, page 1.
- 10:11 (4) Mixer Fire: Naval Propellant Plant, 18 July 1956.
200-gallon J. H. Day mixer. Carlock #234 plain yarn packing rings, five in each gland. Propellant at times contaminates the entire gland. Propellant binder system; nitrocellulose colloids with an ether-diphenylamine mixture. Aluminum panel mixer lid is secured to bowl during mixing. Mixer had been charged with alcohol-wet reground powder and dry mixed for five minutes. Operator who opened valve to admit ether-diphenylamine solvent heard an unusual noise inside mixer and fire was detected. Investigation Held: No definite cause, but most probable cause was due to powder caught between hub of mixer blade and overheated by friction.
Reference 11, page 78.
- 10:11 (5) Mixer Explosion: Aerojet-General, 26 July 1957.
Baker Perkins mixer. An explosion occurred while dry combustion modifiers (carbon black and copper chromite) were being introduced into a propellant mix containing no aluminum and 50% of the oxidizer required for formulation.
Reference 12, page 31.

- 10:11 (6) Mixer Explosion: Aerojet-General, 10 August 1957.
Baker Perkins mixer. An explosion of high order occurred while ammonium perchlorate was being added (80% having been added) to a mix containing aluminum. Mixer and mix station were demolished. Eleven other buildings on the line were damaged. See 21:11 (2).
Reference 3, page 31.
- 10:11 (7) Incident Occurring During Charging of Dry Mixture of Ammonium Perchlorate and Carbon Black into Mixer.
Memo from H. L. Padgett, 14 August 1957, Thiokol, Longhorn Division.
- 10:11 (8) Mixer Fire: Thiokol, Elkton Division, 13 December 1957.
Five casualties. The fire occurred immediately upon restarting the mixer after the addition of the perchlorate and the iron oxide catalyst. The pressure rise was not enough to blow out the aluminum blowout panel in the wall opposite the mixer, and the flame front progressed throughout the entire building.
Reference 3, page 11. Also see Reference 12, page 35.
- 10:11 (9) Mixer Bay, Casting Can Fire: Thiokol, Wasatch Division, February 1958.
Fire occurred in casting can during latter part of deaeration process. The cause is believed to be due to high localized temperature generated on the side of the casting can either by friction between the can wall and a vibrator strap or by high-frequency flexing of the strap.
Reference 3, page 12.
- 10:11 (10) Mixer Detonation: Aerojet General, Fall of 1958.
2,000 lb. capacity Patterson mixer. Polyurethane propellant binder system. Stainless steel lids are fast-opening, air-operated, automatic on internal pressure rise. Eight packing rings each gland; Teflon, graphite, and standard lantern rings lubricated with Dow Corning #44 or Dioctyl Azelate. Repacked on average of 1,000 hours; no contamination of packing gland. Detonation of a 2,000 lb. batch was attributed to metal object reaching the blade area and becoming lodged between paddle and tub. Two prior incidents were attributed to possible gassing or exothermic action internally, and initiated by static build-up in ungrounded portions of system. All ducting, screens, etc., are now bonded together. Hinged-type mixer lids with fast-acting, air-operated cylinders are more desirable. Mixing performed under vacuum and nitrogen purge.
Reference 11, page 83.
- 10:11 (11) Mixer Fire: Naval Propellant Plant, 22 December 1958.
100-gallon Baker Perkins mixer. Nitroglycerine-nitrocellulose colloid, propellant binder system. Normal batch, 250 pounds. Fire occurred in No. 5 mixer bay, Building 704, while processing ABL2014 (hybrid) casting powder scrap. Mixer had been charged 10 minutes prior to ignition with 318 lbs. (wet weight) scrap. No solvent had been added. Thirty to 60 seconds before the fire, an operator heard a noise as though the motor was laboring very hard. Mix ignited before the power could be cut. It is believed ignition was probably due to friction between the powder and the mixer wall. Part of the required amount of acetone needed is now added at the start of the mixing.
Reference 11, page 48.

- 10:11 (12) Mixer Fire: Hercules Powder Company, 28 April 1959.
Readco Tangential Sigma Blade mixer. Double-base propellant. Mixer was being used to process high energy propellant. The mixer had run through the mix cycle and was on chill when the incident occurred. It is generally believed that this fire was the result of material hardening in the glands and that friction from the mixer blades started the fire. Procedure was established whereby personnel would wet gland openings periodically with solvent.
Reference 11, page 31.
- 10:11 (13) Mixer Explosion: Naval Propellant Plant, Indian Head, Maryland, March 1959.
Baker Perkins mixer. Explosion occurred approximately one minute after addition of ammonium perchlorate to the NG/NC base mix. Considerable damage but no personnel injury (operators protected by a 12" reinforced concrete wall). For theory see 21:15 (1).
Reference 2.
- 10:11 (14) Mixer Fire: Thiokol, Longhorn Division, 31 July 1959.
Building 41-G, 200-gallon Baker Perkins mixer. Operator stated a muffled explosion occurred about four minutes after the mixer was turned on. Mixing was by reverse process. Polysulfide/ammonium perchlorate mix. Mixer not damaged; one first-aid case.
Reference S&S File 340-1/304.4.4.
- 10:11 (15) Two Mixer Explosions: Hercules Powder Company, 5 October 1959.
Two 150-gallon Sigma Blade mixers located in adjacent bays were operated simultaneously. Nitrocotton, nitroglycerine, and solvent were placed in the mixer; dry aluminum and dry oxidizer were placed on top. An explosion occurred in the east mixer first, followed within seconds by an explosion in the west mixer. The nature of the damage suggested that a considerable amount of the ingredients must have been consumed as pyrotechnic.
- 10:11 (16) Mixer Fire: Thiokol, Longhorn Division, 22 December 1959.
200-gallon Baker Perkins mixer. Polysulfide/ammonium perchlorate mix. Mixing by reverse process. Mixer had been operating between thirty seconds and one minute when fire occurred. No injuries. Propellant was consumed in fire. Mixer not damaged. Possible Cause: Foreign material introduced in mixer.
Reference S&S File 340-1/304.4.
- 10:11 (17) Mixer Explosion: Thiokol, Longhorn Division, 8 January 1960.
Building 41-G. 200-gallon Baker Perkins mixer. Mixing polysulfide/ammonium perchlorate propellant by reverse process. The mixer had been operating about one minute when the explosion occurred. Building destroyed. No injuries.
Reference S&S File 340-1/304.4.4.
- 10:11 (18) Mixer Fire: Thiokol, Wasatch Division, 6 January 1960.
Reverse mixing (ammonium perchlorate first added). PBAA and aluminum paste added during a 30 minute mix cycle. Baker Perkins mixer. Premix paste consisting of ERL and aluminum was then added. Make-ups (AP and first premix) also added. After a 10-minute mixing cycle, samples were taken and the mixer scraped down. Mixer was re-started, and in about two minutes the fire occurred. Cause of the fire undetermined. Possible Causes: 1) foreign objects in mixer; 2) aluminum and ammonium perchlorate dust and a volatile in the PBAA reacted; 3) the mixer malfunctioned during the final 10-minute mix cycle.
Reference Thiokol, Wasatch Division TU-87-2-60.

- 10:11 (19) Continuous Mixer Incident: Rocketdyne, McGregor, Texas, May 1960.
The mixer consists of an electrically driven propeller, which holds about 15 pounds. Two hoppers are used to feed the mixer; one contains dry materials and the other a "slurry" aluminum powder and Dow Corning "200". The mixer had "bogged down" and they had shut off the machine. Four men entered the bay and were dipping the slurry out of the hopper with a cardboard box. After being in the bay for at least 10 minutes, a small explosion--evidently in the pump housing--threw the slurry in the air and burned all four men in the bay. Only one man out of the four survived.
- 10:11 (20) Fire in 20-Gallon Mixer: Thiokol, Huntsville Plant, 12 July 1960.
Part of fuel screen found in mixer following the fire. Minor injury to an operator. Baker Perkins mixer.
- 10:11 (21) Mixer Fire: Thiokol, Huntsville Plant, September 1960.
Baker Perkins mixer. Fire occurred during cleaning. Parts of detergent screen found in mixer following the fire. No injuries.
- 10:11 (22) Mixer Fire: Hercules Powder Company, 14 September 1960.
Readco Tangential Sigma Blade mixer. Double-base propellant. No. 2 mixer in Building 3673 fired while processing high-energy propellant. The mixer had completed cycle and had been turned down for pulling. The operator proceeded to unlatch the lid (lid is cast iron with lead blow-out panels and is counterbalanced and held by shear ring arrangement on all but composite multiple double-base mixers which are not counterbalanced but held by air cylinders) and the fire occurred. It is believed that friction could have caused this incident. Preventive measures were taken and the catch was removed from the lid; lid hinge pins were equipped with Teflon washers; counterweight cable was moved to provide clearance between mixer and cable.
Reference 11, page 31.
- 10:11 (23) Continuous Mixer Incident: Thiokol, Longhorn Division, 9 December 1960.
Building 68-G, no injuries. Ignition occurred somewhere along the screw assembly, which resulted in sufficient pressure build-up to shear the bolts holding the mixer section together. For possible cause, see 21:15.
- 10:11 (24) Continuous Mixer Incident: Thiokol, Wasatch Division, 6 January 1961.
Continuous mixer (Ko-Kneader) incident occurred right after the mixer had been started up after being "bogged down". The screw had been backed up, the mixer re-started, and had made two revolutions (in connection with its forward motion) when the incident occurred.
- 10:11 (25) Mixer Fire: Hercules Powder Company, 6 April 1961.
Readco Tangential Sigma Blade mixer. Double-base propellant. Mixer No. 2 in Building 3692 had completed the mix cycle and had been drying for a short period. The operator had added additional solvent, closed the lid, and left the building when the fire occurred. No apparent reason for the fire could be found.
Reference 11, page 31.
- 10:11 (26) Mixer Explosion: Thiokol, Longhorn Division, 13 July 1961.
200-gallon Baker Perkins mixer. Forward mixing. Near completion of mix, some 60 seconds after all the oxidizer had been added, an explosion occurred. Probable Cause: Initiation in packing gland.
Reference S&S Department Report of 50-B Incident.

- 10:11 (27) Mixer Fire: Hercules Powder Company, 20 August 1961.
Readco Tangential Sigma Blade mixer. High-energy propellant. Mixer No. 1 in Building 3692 had been charged with remix high energy propellant and had been on cycle about 40 minutes when a fire occurred. It is believed the fire was the result of a press screen being charged into the mixer with the remix material. Remix material is now passed over a metal detector before being put in the mixer.
Reference No. 11, page 31.
- 10:11 (28) Mixer Fire, Outside Source of Ignition: Naval Propellant Plant, 10-5-61.
150-gallon, L. H. Day Company, Propellant Binder System. Nitroglycerine - nitrocellulose colloid. After the mix cycle, and prior to discharging mix, an operator attempted to remove the catch pan (used to catch propellant which passes through the annular space during mixing) and on seeing a flame originate inside the pan, dropped it and ran. The flame spread and ignited the propellant in the mixer, but was soon extinguished by activation of the sprinkler system.
Reference 11, page 71.
- 10:11 (29) Mixer Fire: Thiokol, Huntsville Division, 9 February 1962.
During addition of ammonium perchlorate to a 200-gallon Baker Perkins mixer, a fire occurred. Cause of the fire believed to be due to spilled oxidizer near frozen oil contaminated pump shaft coupling, which ignited by friction of rotating motor coupling.
Report No. U-A-62-4238A.
- 10:11 (30) Mixer Fire: Rocketdyne, McGregor, Texas, 22 June 1962.
Ammonium nitrate propellant fire occurred in a 100-gallon Baker Perkins mixer. Cause not stated. Only minor damage done due to operation of vacuum-stat which raised the mixer lid permitting automatic operation of the water deluge which extinguished the burning propellant.
Reference Telegram, RT-1188, Safety File 1/304.4.6.
- 10:11 (31) Vertical Mixer Fire: Thiokol, Elkton Division, 7 September 1962.
Baker Perkins vertical mixer, 150-gallon capacity. Mixing had been in progress for 38 minutes when a fire proceeded by a "boom" occurred. It is believed a three-piece plug (consisting of a neoprene doughnut around an aluminum insert, into which is placed a thermocouple) had, as a result of mixing action, unscrewed and drifted upward and was struck by the blades. Score marks were found on the aluminum portion of the plug, as well as on the bottom of the mixer blades. Damage was light due to: 1) quick action of the deluge; 2) failure of the hydraulic system (possibly after ignition) permitting bowl to be thrust to the floor away from the superstructure. Future design may incorporate burst diaphragm into hydraulic cylinders. Down time 10 days.
Reference S&S File 340-1/304.4.6.
- 10:11 (32) Mixer Fire: Hercules Powder Company, 13 November 1962.
Readco Tangential Sigma Blade mixer. Triple-base propellant. No. 2 mixer at Building 3608 fired while processing triple-base propellant. The operator entered the bay to check the mixer and when he started to throw it out of gear, the fire started in the right front gland. No apparent cause of the fire was found.
Reference 11, page 31.

- 10:11 (33) Mixer Explosion: Atlantic Research Corporation, 3 January 1963. 100/150 DA chemical mixer, 1,350 lbs. normal batch, propellant - PVC composite. Mixing done under vacuum. Ground NH_4ClO_4 is not screened. Mixer was destroyed in an explosion while being operated remotely. There were no injuries to personnel. Findings to be reported later.
- 10:11 (34) Mixer Fires: Rocketdyne, Solid Propellant Division, Year 1956 to 20 June 1963. 100-gallon Baker Perkins mixer used for mixing of ammonium-nitrate, rubber-base extrudable propellant. Normal batch, 450 to 525 pounds. Six rings of packing (Garlock #1100 braided asbestos impregnated with graphite) used in each packing gland. Propellant contamination extends all the way through the packing gland assembly to the packing followers. There have been six fires in the seven-year period. Each fire damage was minor. See 21:15 (12). Reference No. 11, pages 64 to 67.
- 10:11 (35) Mixer Explosion: Atlantic Research Corp., Alexandria, Va., 2 January 1963. A READCO horizontal mixer was being used to mix approximately 1,300 lbs. of an aluminized composite propellant containing ammonium perchlorate. All ingredients had been incorporated into the mix and a 20-minute mix cycle under vacuum had been completed. Following this, the mixer was to be operated in reverse direction to clean blades. The explosion occurred at the instant the switch was thrown to start the reverse operation. Specific cause of the ignition is not known. The mixer building was completely demolished and several secondary structures were severely damaged. Glass breakage was reported at distances up to $1\frac{1}{2}$ miles.
- 10:11 (36) Mixer Explosion: Thickol, Wasatch Division, 23 August 1963. A confined deflagration occurred in a 300-gallon Baker Perkins horizontal mixer located in Building M-25 in the R & D Area. The mixer involved was recently converted to the automatic batch weighing mix system. The incident occurred after 30 minutes of the mix cycle. The oxidizer and premix additions were completed. Minuteman propellant was involved. No injuries.
- 10:11 (37) Vertical Beken Mixer Fire: Rocketdyne, McGregor, Texas, 6 September 1963. Propellant ignition occurred in a 25-gallon vertical Beken Mixer. There were no injuries. A batch of conventional ammonium perchlorate castable propellant was being mixed. The point in the mixing cycle had been reached for attendant addition of MAPO curative. Temperature of mix was approximately 176°F . Mix can had been lowered, sides of mix can scraped down, and the undiluted MAPO distributed over the top of the propellant mix. Operator reported that there was no visible unincorporated ammonium perchlorate present at the time of MAPO addition. Approximately 2 to 4 minutes after MAPO addition, spontaneous ignition occurred. Complete action of deluge system limited damage to scorching of the building walls and scorching of the mixer. Probable cause of ignition thought to be exothermic reaction between MAPO and ammonium perchlorate.
- 10:11 (38) Continuous Mixer Fire: Thickol, Wasatch Division, 18 December 1963. A fire and subsequent pressure burst occurred in the Ko-Kneader section of the composite solid propellant continuous mixing facility. The fire, propagated from the Ko-Kneader section to the associated equipment in the mixing room, then went through the analytical room to the east exterior of the building and into the propellant waste receivers. \$271,000 damages, no injuries. The Board of Investigation concluded that heat of friction created by the entrapment of an unidentified metallic object between the outer edge of the mixer screw flight and the mixer barrel, precipitated the incident. Reference Report of Board of Investigation, Composite Solid Propellant Continuous Mixer Fire, Thickol, Wasatch Division, 18 December 1963.

MOTOR CASTING, CURING, AND FINISHING

- 10:12 (1) Motor Cut-back, Nike-Zeus: Grand Central, December 1960.
While using a manual mechanical advantage-type cutter for propellant trimming, the motor was ignited, killing one operator and injuring two others. It is believed dust and small particles of propellant could have been ignited due to one or more of the following causes: 1) mechanical friction, 2) pinching of propellant between two hard surfaces, 3) static discharge between personnel and propellant, 4) metal object struck a spark.
Thiokol Safety Section File (Longhorn) 341-1/304.4.6.
- 10:12 (2) Motor Cut-back, LaCrosse: Huntsville Division, 16 April 1957.
An ignition occurred during the remote cut-back operation. A second engine, bolted to another stand in the same bay, ignited five or six seconds after the first. The fire was probably caused by the propellant between the end of the cutter blade and the casting sleeve being ignited by impact or friction. The possibility of tramp metal in the propellant was considered, but seemed unlikely. For corrective action, a more rigid preventive maintenance program was started. Clearance between blade and case should be increased.
Reference 3, page 5.
- 10:12 (3) M-58 Motor, Cut-back Fire: Longhorn, 7 July 1956.
During cut-back of an M-58 motor, using a disk blade operated by remote control, a fire occurred. No injuries due to the motor being secured in a steel cubicle vented to the outside.
Reference Safety Section File (Longhorn) 341-1/304.4.6.
- 10:12 (4) Dropped Loaded Motor: Building 54-F, Longhorn, 19 October 1957.
A loaded motor ignited and burned when dropped in an oven room. The motor dropped due to a hoist failure.
- 10:12 (5) Accidental Firing of a T-131 Rocket Engine: Huntsville, Alabama, May 1953.
The incident occurred when an oversized nozzle plate was being forced, by use of an impact wrench, into the internal engine threads. The igniter was in the engine, the operator was unshielded, and the engine was held in a vise that was not anchored to the workbench. Three operators were involved.
Reference 3, page 2. Container 108 - Longhorn.
- 10:12 (6) Motor Cut-back Ignition, Burning, and Detonation: Thiokol, Reaction Motors Division, 10 February 1961.
TDX-2 (Series G) solid rocket propellant—about 90% cut had been made. After ignition of the motor, it burned for a moment with one end closed by the saw blade. The hold-down straps failed and the motor stood up vertically and detonated.
Memorandum from James F. Hayes, Manager, Safety and Security Department, Reaction Motors.
- 10:12 (7) Casting Bay Incident: Huntsville Division, 6 October 1953.
The rocket propellant was loaded into the rocket motor body by using a screw type horizontal extrusion press. The body of the motor has three threaded vent holes which have to be cleaned after the motor is filled and removed from the extrusion press. After cleaning, threaded metal vent plug-stoppers are screwed into the vent holes. While one of the operators was engaged in tightening one of the vent plugs, an explosion occurred. The flame from this explosion ignited the propellant remaining in the feed hopper, feed screw, and in the adapter sleeve of the extrusion press. The four men in the press bay died. Abstract Report No. E. S. 635, 6 October 1953, Department of the Army, Office of the Chief of Ordnance. Longhorn S&S File 340-1/304.4.6.

- 10:12 (8) Motor Cut-back, 12,000 Pounds: 6 September 1961.
Explosion occurred involving a Wing 1 conventional solid propellant rocket motor. During cut-back operations and pre-operation of the cutting device, sparks were noted which subsequently ignited the grain of the 12,000 pound acle model motor.
Reference A3ESB Explosive Incident Report No. 999.
- 10:12 (9) Curing Oven Incident: Thiokol, Longhorn Division, 30 March 1962.
Operators believed to have heard a metallic noise or clang inside the bank of ovens, and a short time later observed propellant flowing across the oven bay floor, which ignited. Within a few seconds, an explosion took place which was followed by two others at close intervals. Four XM-30's in all were involved in the incident. Explosion of the motors may have been the result of internal ignition of the propellant grain from external heat. Probable Cause: The head-end retainer ring came loose on a freshly cast motor that had been suspended in the oven an hour previously. This permitted the head-end casting assembly to drop onto the expanded metal cover over the fan and heater assembly beneath the suspended motor. Propellant then could flow out the head-end of the motor onto the fan and heater. Propellant on the depressed expanded metal cover may have been ignited by the fan blade striking the metal cover or head-end assembly.
Reference Incident Report, Building 54-F, 30 March 1962.
- 10:12 (10) Curing Oven Incident: Hercules Powder Co., Bacchus, Utah, 23 August 1962.
Three or four thousand pounds of high-energy propellant in a cure house violently exploded while three men were in the cure house to get some small test motors. It is believed the incident was caused by dropping one of the test motors. All three men perished.
- 10:12 (11) Casting Bayonet Incident: Thiokol, Wasatch Division, 23 March 1962.
Fire started when an operator placed the flanged end of a large propellant-filled casting bayonet on the concrete floor with too much force. Cause of fire, either friction or sparks. No one injured; damage \$6,500.
- 10:12 (12) Fire in Blocking Press: Astrodyne, Inc., McGregor, Texas, 21 December 1956.
No injuries. \$924 damages. Possible Causes: 1) compression of air in press due to blocking of vacuum port to ram or due to entry of air into basket because of defective O-ring, 2) foreign material in propellant.
Reference 12, page 10.
- 10:12 (13) Extruder Deflagration: Astrodyne, Inc., McGregor, Texas, 1 March 1956.
Three injuries. \$7,200 damages. Possible Causes: 1) heating of propellant above its auto-ignition temperature by compression of entrapped air, 2) basket of press was overloaded and blocks were being forced into basket by use of ram.
Reference 12, page 9.
- 10:12 (14) Explosion of Extruder: Astrodyne, Inc., McGregor, Texas, 13 April 1957.
No injuries. \$395,000 damages. Possible Causes: 1) compression of entrapped air, causing propellant to heat above its auto-ignition temperature, 2) mechanical failure of extruder components, permitting metal-to-metal contact, 3) frictional heat generated by rubbing ram against barrel of press, 4) impurities in propellant, 5) friction of screw from vacuum post in ram between ram and barrel of press. 240 pounds AP propellant involved. Recommendations: 1) provide automatic vacuum and pressure recorders on extruders, 2) provide automatic stop on ram in event of vacuum loss.
Reference 12, page 11.

- 10:12 (15) Ignition of Propellant on Casting Can Lid Clamp: Thiokol, Longhorn Division, 29 August 1963.
During assembly of M-30 casting can, a small amount of propellant ignited between the lid clamp and assembly nut while the casting can lid was being secured to the casting can. The incident occurred when operator began tightening the lid clamp assembly bolt nut. A small flash and some smoke comparable to a lighted match occurred between the clamp and bolt nut. The lid clamp was removed from the can and thoroughly cleaned with solvent. It was then installed and the casting operation completed.
Longhorn Safety Section File 341-1/304.4.5.
- 10:12 (16) See 10:11 (9) - Casting Can Fire in Mixer Bay, resulting from heat generated by vibrator.
- 10:12 (17) Ignition of Motor During Cure: Grand Central, 7 July 1956.
One fatal injury. Oven and cell destroyed. 1,250 lbs. of polysulfide perchlorate propellant involved. Inspector was checking oven temperature when motor ignited. Cure temperature was 190°F in pit-type oven with 30 psi nitrogen on motor. Source of heat was a hot water heat exchanger operating at 210°F. Highest possible exotherm existing in propellant during cure is believed to be 220°F. Motor caught fire after eight hours cure. Mandrel was ejected 200 feet. Propellant could not be ignited below 388°F in Wood's metal bath.
Reference 12, page 45.
- 10:12 (18) Propellant Fire in Electric Oven: Astrodyne, McGregor, Texas, February 1954.
One injury. \$18,400 damages. 1,080 lbs. of ammonium nitrate propellant in Cell No. 2 at time of fire. 395 lbs. were in Oven 2 where fire started.
Possible Causes: 1) propellant touching baffle plate (between oven and calorimeter units), causing ignition, 2) local overheating of oven.
Reference 12, page 17.
- 10:12 (19) Ignition of Motor During Cure: Grand Central, 2 October 1956.
Two fatalities. Four curing ovens destroyed. Polysulfide ammonium perchlorate propellant. 1,250 pound motor ruptured. Cure temperature was 180°F under 30 psi nitrogen. Possible Causes: 1) possible exothermic heat from curing and low auto-ignition temperature of the propellant, 2) mandrel may have expanded lengthwise, compressing propellant in mandrel centering cup.
REMARKS: a) Exotherm from a non-homogeneous fuel-rich portion of propellant could be considered higher than the average propellant. The adiabatic maximum temperature of the fuel alone cured at 180°F has been measured at 321°F, and at 190°F to be 340°F (corrected for calorimeter heat absorption would raise reading about 30°F); b) Propellant auto-ignition temperature was 393°F at four plus minutes (Wood's metal bath); c) In a sample 240-pound propellant charge, the maximum temperature rise was 30°F in an oven at 190°F after about eight hours, and a maximum rise of 22°F was attained in thirteen hours at 180°F (exothermic reaction); these times correspond closely with the ignition times in this incident and the one on 7 July 1956, d) From information received on test run to determine auto-ignition temperatures on this propellant with various additives included, copper chromite may have been an ingredient in this propellant formulation. On tests which were performed, propellant containing copper chromite exploded on ignition.
Reference 12, page 46.

10:12 (20) Ignition of Propellant During Cure: Jet Propulsion Laboratory, 1953.
Several solid charges of 2-inch diameter propellant ignited during cure.
Possible Cause: Addition of 4% ammonium dichromate to ammonium nitrate-GG
(Glycidyl Glycerin) propellant lowered the auto-ignition of the composition
to 130°C; consequently, this low ignition temperature, combined with a
moderate exotherm, caused ignition. Remarks: Ammonium dichromate attacks
the resin binder and causes heat to build up. Recommendation: Manganese
dioxide proved a satisfactory replacement for ammonium dichromate.
Reference 12, page 58.

OXIDIZER INCIDENTS (also see 12:11 and 20:10)

- 10:13 (1) Oxidizer Grinding: Thiokol, Huntsville Division.
A canvas adapter sleeve, used to conduct oxidizer to a drum, caught fire. The cause was apparently due to heat generated by friction between the sleeve and a drive belt.
Reference 3, page 2.
- 10:13 (2) Drum of Ammonium Perchlorate Ignited: Thiokol, Huntsville Division.
When unloading a drum of ammonium perchlorate, one of the drums was ignited and was propelled into a workman. Later tests established that enough heat or friction could have been generated by the sliding of the metal drum down the metal skid to ignite the polyethylene liner in the drum.
Reference 3, page 2.
- 10:13 (3) Bumped Gyrotory Screen Against Wall: Longhorn Division, 10 June 1959.
This caused the ignition of the perchlorate impregnated sponge rubber gasket around the edge of the screen frame, which in turn ignited the perchlorate impregnated canvas on the screen. No injuries.
Reference Longhorn Safety File 341-1/304.4.4.
- 10:13 (4) Two Drums of Ammonium Perchlorate Detonated: 15 February 1960.
Two drums of ammonium perchlorate detonated in a mixer bay as a result of a mixer explosion. It is believed their detonation was due to high velocity particles rather than the detonation wave front from the mixer explosion.
See 12:12, also 12:11 (5).
Reference Minutes of T&SC Meeting, Longhorn Safety & Security File.
- 10:13 (5) High-Energy Oxidizer, Laboratory Incident: Thiokol, Huntsville Division, September 1960.
During an attempt to prepare high-energy oxidizer materials (laboratory quantities - N_2F_4 and C_6H_6), a chemical exothermic flame-off occurred while vacuum distilling the reaction products.
- 10:13 (6) HP₂ Explosion in Storage Shed: Thiokol, Wasatch Division, 9 September 1963.
Individual quantities of HP₂ in various process stages were stored in a thick polyethylene bag inside a polyethylene container inside a 30-gallon drum. The drums were stored on a pad under an open storage shed. Burning preceded the explosion by an estimated 15 seconds. The most probable cause was an exothermic reaction in one of the containers—possibly between HP₂ and the epoxy painted drum. Extensive stability tests were run and valuable recommendations made for safer storage.

CHEMICALS AND OTHER MATERIAL - PYROTECHNICS

- 10:14 (1) Paraquinone Fire: Longhorn Division, 23 October 1956.
When using an ungrounded vacuum hose to clean up spilled paraquinone dioxime, a fire resulted, and it is believed that the cause of the ignition was the result of a discharge of static electricity built up in the hose.
Reference Safety Section File 341-1/304.4.4.
- 10:14 (2) Pyrotechnic Mixer (Muller Type) Fire: Thiokol, Wasatch Division, December 1959.
A mixture of magnesium, aluminum alloy, potassium perchlorate, and potassium nitrate were being blended in a small Muller mixer. The binder was to be left out until the fuels and oxidizers were completely mixed. The above dry ingredients had been mixed for five minutes; then the mixer shut down and an operator entered the mixer bay (after a five-minute waiting period so that no static charge or delayed action would take place while the operator was in the bay) to check the mix for complete mixture, and brush (bristle) the unmixed oxidizer from around the outer edge of the mixer back into the mix. The mixer was restarted and after making about two revolutions, the mix ignited. Apparent cause was due to sparks or heat from metal-to-metal contact of steel mixer blades with the steel mixer body.
Incident Report, Pyrotechnic Deflagration, Thiokol, Wasatch Division.
Longhorn Safety & Security Dept. File No. 340-1/304.4.6.
- 10:14 (3) Diborane Surge Tank Explosion: 15 March 1961.
A 2,000 cubic foot tank containing 1,775 pounds of diborane involved.
Rupture of the one-inch steel tank was believed to be due to a defective weld.
ASESB Report No. 32.
- 10:14 (4) Explosion in Tumbling Barrel Containing Pyrotechnic Mix: Thiokol, Hunter-Bristol Division, 17 May 1961.
- 10:14 (5) Methyl Bromide Tank Explosion: Explosive limits are greater than once believed. Zinc, aluminum, and magnesium are known to form pyrophoric grignard type reaction products. Therefore the term "non-ferrous metals" should not be used in reference to storage containers.
ASESB Incident Report No. L-28.
- 10:14 (6) Trichloroethylene has been regarded as a non-flammable solvent, but recent tests by the Bureau of Mines prove otherwise. See "Flammability of Trichloroethylene", Bureau of Mines Investigation Report 6190.
- 10:14 (7) Ammonium Dichromate Fire: Astrodyne, McGregor, Texas, September 1955.
Approximately 150 pounds involved in a fire during grinding operations.
Possible Causes: 1) static spark between blade and falling dichromate, 2) V-belt overheated, 3) over-heating of air lock valve Teflon blade.
Corrective Action: control dust; provide electrical grounds.
Reference 1', page 26.
- 10:14 (8) Milori Blue Fire in Dryer Oven: Astrodyne, McGregor, Texas, February 1956.
Approximately 180 pounds involved. Decomposition temperature not determined at this time. Possible Causes: 1) prolonged heating (for 113 hours at 225°F) of Milori blue (iron blue) may have caused decomposition and ignition, 2) contamination, 3) catalytic reaction caused by moisture from bottom of barrel passing through dry (drier) blue at top of barrel.
Reference 1', page 20.

CONTAMINATED VACUUM LINES AND EQUIPMENT; STATIC ELECTRICITY, EXPLOSIVE GASES

- 10:15 (1) Explosion in Vent Stack - Static Generation: Employee, after oiling a fan shaft, turned on the electric motor switch. Fan failed to revolve so employee (wearing street clothes underneath synthetic fiber coveralls) reached down into vent stack (containing methyl alcohol and benzene vapors) to give the fan a turn. An explosion occurred, burning employee on face and hands. ASESB Explosive Incident Report No. L-34.
- 10:15 (2) Toluene - Static Sparks: While toluene was being transferred by hand pump from a drum to an ungrounded pail, static sparks jumped from the nozzle to the pail, igniting the toluene. The nozzle was dropped and toluene syphoned from the drum to the floor of the main work area. Burning liquid flowed under the fire door (no curb provided) into a mixing room. ASESB Explosive Incident Report No. L-6.
- 10:15 (3) Polyethylene Liner - Static Fire: After scooping dry chlorothiaside intermediate (DSA-chlorodisulfany aniline) from a polyethylene lined fiber drum into an agitated still containing tetrahydrofuran, the operator removed the polyethylene liner and shook the remaining material into the vessel. When shaking the liner, a sudden flash occurred. Relative humidity 50 per cent. Note: Possibility when shaking material like GMP, nylon, etc., from a bag. ASESB Explosive Incident Report No. L-7.
- 10:15 (4) Decaborane Fire, Polyethylene Bag - Static Charge: A technician had removed a scoopful of decaborane from a drum and emptied it into a polyethylene bag on a work table approximately 1½ feet from the drum. He heard what he describes as a "snap" or "crackling" noise as the material in the bag burst into flame. The fire spread to the drum and ignited it. Cause: It is believed that a static charge built up on the scoop and discharged to the polyethylene bag, passed through the decaborane dust in the bag and ignited it. ASESB Explosive Incident Report No. L-31.
- 10:15 (5) Ignition of Igniters in Polyethylene Bag: Thiokol, Huntsville Division, March 1962. Twenty-five igniters are placed in a 6" polyethylene bag. The bag is sealed and bags are labeled with a felt pen. Two men were burned as a result of ignition of the igniters. It is believed the operators had proceeded to the point where some of the bags were being marked when the incident occurred. Exact cause of the ignition is not known. Telephone Conversation, Safety & Security File No. 340-1/304.4.6.
- 10:15 (6) Explosion of Vacuum Line Servicing Propellant Sample Saw: Thiokol, Huntsville Division, 18 June 1962. During sawing of H-Series propellant sample, a dust vacuum line exploded. One operator was killed by fragments from the heavy metal line. Another employee sustained deep cuts in the hip, and a third employee sustained first-degree burns during rescue work. Exact cause of explosion unknown at time of report. Reference Safety & Security File No. 340-1/304.4.6.

LABORATORY INCIDENTS: SAWING, MIXING, HEATING

- 10:16 (1) Sawing Propellant Sample: Thiokol, Longhorn Division, 1955.
When sawing a propellant sample with a commercial type band saw, the propellant ignited and burned. It is believed the cause of ignition was due to build up of heat by excessive linear speed of the band saw. Standards for the set, rake, and speed of the saw have now been established.
- 10:16 (2) Sawing Propellant Sample: Thiokol, Huntsville Division, 19 March 1959.
Two operators were engaged in sawing samples when the propellant ignited and burned. It is believed heat from friction between the band saw blade and blade guide caused the ignition.
- 10:16 (3) Sawing Propellant Sample: Thiokol, Longhorn Division, 1959.
After an operator had cut some propellant samples with a commercial type band saw (approved electrical class), he attempted to clean the blade with a brush. Propellant cuttings beneath the saw table became ignited and the fire spread to nearby samples. It is believed the cause of the fire was undue pressure against the band saw which ignited grains of propellant between the band saw and saw guide.
- 10:16 (4) Explosion of Vacuum Line Servicing Propellant Sample Saw: See 10:15 (6).
- 10:16 (5) Lithium Aluminum Hydride/Ether, Fire: Chemist was pouring ether (which had been dried over calcium hydride) into a flask containing lithium aluminum hydride. A flash occurred burning the chemist.
ASESB Explosive Incident Report No. L-4.
- 10:16 (6) Hand Cutting Sample from 92-Pound Motor - Ignition: Rocketdyne, McGregor, Texas, 5 July 1962.
When using a beryllium alloy knife to cut a laboratory test specimen from a 92-pound cast ammonium perchlorate motor, the operator noticed sparks at the point of cut. The grain burned rapidly, but the operator escaped with only a slight burn. Forty previous samples had been taken in a similar manner without incident. Cause believed to have been due to friction inherent in the cutting action.
Manufacturing Chemist Association, Inc., October 1962 issue.
- 10:16 (7) Metal X Fire in Laboratory Hood: Thiokol, Huntsville Division, 26 Nov. 1963.
3-400 grams sample involved. Following the fire, extensive atmospheric tests were run before personnel were permitted in vicinity of laboratory.
- 10:16 (8) Laboratory Oven Fires: Astrodyne, McGregor, Texas, Years 1956 and 1957.
Possible causes of five different laboratory oven fires:
- 1) Igniters were to be cured for two to four hours at 170°F. Operator set oven at 168°C, and igniters caught fire on reaching auto-ignition temperature. Corrective Action: Select one temperature scale for use in oven room.
 - 2) Heater relay contact stuck, permitting oven to overheat, resulting in propellant ignition at 365°F. Corrective Action: Functional check at least weekly.
 - 3) Auxiliary heater switch was mistakenly left in the "on" position, permitting oven to overheat and ignite propellant. Correction Action: Replace toggle switch with one that cannot be inadvertently activated.
 - 4) Low ignition temperature of ammonium nitrate propellant containing EL Chemical 1834.

- 5) Probable short circuit in oven or control due to deluge water in oven, causing oven to overheat and propellant ignition. Corrective Action: After deluge and before start-up of electric oven, check dryness.

Reference 12, Pages 21, 22, 23, and 24.

- 10:16 (9) Beryllium Hydride Incident: Thiokol, Reaction Motors Division, 13 Dec. 1963. Laboratory hood fire involving small amount of BeH_2 in a high-energy fuel. Reference TLD No. O-63-1890.

TRANSPORTING RAW PROPELLANT, MOTORS, ETC.

- 10:17 (1) Overturning of Casting Can - Ignition of Propellant: Thiokol, Longhorn Division, August 1962.

A casting can containing 2,400 pounds of raw, XM-30 propellant, "toppled" from the dolly on which it was being transported as the tow motor operator was executing a right turn. When the can struck the pavement, the propellant ignited and burned. The operator had failed to secure (by use of pins) all four legs of the casting can in the "wells" provided on the dolly.

- 10:17 (2) Transporting Emptied Casting Can and Casting Hose: Thiokol, Wasatch Division, 1961.

When transporting an emptied casting can and casting hose on a dolly to the cleaning building, the tow motor operator looked back and observed that the flanged end of one of the casting hose was dragging on the pavement. Sparks were noted, and at the same time propellant in the hose ignited. All hose on the dolly burned. Propellant remaining in the casting can did not ignite.

CLEANING PROPELLANT EQUIPMENT

(Cleaning Degreaser - See 20:13)

- 10:18 (1) Casting Can Cleaning Incident: Thiokol, Huntsville Division, 30 August 1961. Operator was in a crouched position inside a 300-gallon casting can scraping the remaining propellant loose with a stainless steel spatula. The can is placed in a horizontal position for cleaning. Operator saw a glow on the tip of his spatula an instant before a fire occurred which hurled him approximately 20 feet. Operator was off work 49 days. Reference Thiokol, Huntsville Division, "Fire During 300-Gallon Casting Can Cleaning Operation", Control No. U-A-61-4254A.
- 10:18 (2) Casting Bayonet Incident: Thiokol, Wasatch Division, 23 March 1962. Fire started when an operator placed the flanged end of a propellant-filled casting bayonet on a concrete floor with too much force. Cause of fire was either friction or sparks. No one injured.
- 10:18 (3) Cleaning Casting Tube: Thiokol, Huntsville Division, 22 May 1961. An operator was cleaning an offset casting tube assembly which consisted of two 6" x 24" marman flanged straight sections and two marman flanged elbows. The excessive propellant was being removed through the assembly by forcing a clean cloth ball through the tube with 80⁺ psi of air pressure. A fire occurred when the ball came out of the tube. Investigation showed a female elbow and female straight joint were assembled together and pinching could have occurred when the pressure released. One first-aid case.
- 10:18 (4) Cleaning 300-Gallon Casting Can: Thiokol, Huntsville Division, 8 Sept. 1961. Fire occurred while an operator was cleaning waste propellant from a 300-gallon casting can with a stainless steel hand spatula. Possible Cause: Undue pressure applied to spatula while removing propellant from the bottom section of the casting can.
- 10:18 (5) Mixer Cleaning: Thiokol, Huntsville Division, 17 October 1959. A flash fire occurred during mixer cleaning operation while using sawdust and detergent as the cleaning agent. Cause: Foreign body accidentally introduced into the mixer. A similar incident occurred on 16 January 1959 when a piece of tramp metal was introduced into the mixer with the sawdust. On 21 September 1960, a screen used to screen detergent into the mixer was accidentally introduced into the mixer during cleaning operations, and as a result, a fire occurred.
- 10:18 (6) Motor Case Cleaning: Thiokol, Huntsville Division, 9 December 1958. In order to reclaim motor cases, loaded reject motors are soaked in a solvent until propellant softens. An operator using an improvised tool, which had just been sharpened on a grinding wheel, attempted to remove softened propellant from a motor case, and the propellant ignited. It is believed the temperature of the tool was sufficient to ignite the propellant. As a result, several motors were ignited.

TEST FIRING

- 10:19 (1) Explosion of Motor During Test Firing: Astrodyne, McGregor, Texas, June 1957. No injuries. Damage \$27,000. Possible Causes: Igniter blast or extremely high burning rate of igniter caused propellant breakup. Large burning area due to propellant breakup resulted in pressures sufficient to cause failure of case. 3,000 pounds of ammonium nitrate propellant and one 1,200 gram B141 igniter involved in this incident. Reference 12, page 13.
- 10:19 (2) Explosion of Motor During Test Firing: Astrodyne, Inc., McGregor, Texas, October 1957. No injuries. \$52,508 damage. 3,800 pounds ammonium perchlorate propellant. Possible Causes: Failure of propellant-to-propellant, restrictor and/or case bonds, creating excessive propellant burning area and plugging of the forward section of the grain port, resulting in excessive pressure and explosion. Reference 12, page 12.

BURNING GROUND

- 10:20 (1) Explosion During Burning of Scrap Explosives: Astrodyne, Inc., McGregor, Texas, January 1957. Approximately 40 pounds of ignition material and 4,000 pounds of ammonium nitrate and ammonium perchlorate were on burning pad and burned five minutes before exploding. Five craters were formed up to three feet deep and three feet wide. Approximately 500 pounds of unburned propellant was recovered; one piece weighing about 25 pounds was found one-fourth mile away. Possible Cause: Igniter materials confined in cartons exploded due to intense heat of burning waste. Reference 12, page 14.
- 10:20 (2) Premature Ignition of Waste Perchlorate Pad: Thiokol, Longhorn Division. A pad of waste ammonium perchlorate had been prepared for later burning by spreading the perchlorate over sawdust and then pouring oil over the perchlorate. As the workmen were walking away from the pad to prepare other pads of waste, the perchlorate pad ignited. Possible Cause: Accidental contamination of the ammonium perchlorate with a small amount of metal powders which reacted with moist air producing hydrogen gas and heat.
- 10:20 (3) Explosion, Burning Pad: Astrodyne, McGregor, Texas, February 1957. Material had been burning three to four minutes containing 4,000 to 5,000 pounds of ammonium nitrate and ammonium perchlorate propellant. Crater formed was 2 feet deep by 24 feet across. Propellant fragments were projected to one-half mile. Possible Causes: 1) Combined weight and depth of explosive bed might have provided confinement sufficient to initiate explosion; 2) Scrap other than propellants might have exploded, initiating the propellant. Reference 12, page 15.

PROPELLANT INGREDIENTS

ALUMINUM: FIRE AND EXPLOSIVE HAZARD

- 11:10 (1) Aluminum Powder Hazards - Reynolds Metal Company.
Longhorn Safety Section File 341-11/206.1.
- 11:10 (2) Safety Bulletin No. 60.
Thiokol, Longhorn Division.
- 11:10 (3) Milton Cutler, U. S. Army Chemical Warfare Laboratories: "Pyrotechnics as Propellants and Fuels". Aluminum and other metal powders. Handling, extinguishing, etc.
Reference 6, pages 24 - 34.
- 11:10 (4) Atomized aluminum powder may be stored when in sealed containers at 0° to 110°F and 25% to 75% humidity. Outdoor storage not recommended. Drums heated to 135°F should have the air vent screws removed before heating. May require several days for contents of the drum to reach this temperature. Reference is made to a Bureau of Mines Report, "Inflammability and Explosibility of Metal Powders", and a report by Hartman in "Industrial and Engineering Chemistry" on physical test. Sealed drums in storage not required to be grounded. Recommendation on control of atmospheric concentration of aluminum powder, cleaning equipment, and wet type dust collectors. Letter from R. H. Frederick, Regional Industrial Engineer, Powder and Paste Products, Reynolds Metal Company, 4900 Oak Street, Kansas City, Missouri. Addressed to Mr. Ed Brasch, Thiokol, Longhorn Division, dated 24 April 1961.

ALUMINUM: INHIBITED POWDER

11:11 (1) Aluminum powder considered inhibited by treatment with 1% aqueous solution of sodium fluorobate (NaBF_4)—stored in closed containers, ruptured 75 days later.

Thiokol, Huntsville Division, Alabama.

Thiokol Longhorn Safety & Security Container No. 108.

ALUMINUM : THERMITE REACTION WITH Fe_2O_3 , SPARK PRODUCING

- 11:12 (1) Steel, coated with aluminum paint, can emit sparks when struck with a metal striker (steel, brass, or non-sparking alloys) and such sparks may ignite mixtures of flammable gases or vapors. The sparks are due to the thermite reaction between iron oxide and aluminum, and are therefore produced only in the presence of iron oxide. The extent of this hazard depends upon the nature of the surface covered and the nature of the paint. "Accident Prevention Manual", Third Edition, Page 30:17 (several references also given).
- 11:12 (2) Aluminum and aluminum containing alloys are avoided in plant where possible because of the thermite-type reactions with rust or chromium sesquioxide. Explosive Research and Development Establishment. Reference 3, page 48.
- 11:12 (3) See 20:14 (1).
- 11:12 (4) Sparkling Hazard of Aluminum-Painted Iron and Steel Surfaces: In view of the potential sparking hazard, this office concurs that aluminum paint should not be used on ferrous metal surfaces in hazardous locations as defined in Paragraph 603e (1), AMCR 385-224 (ORDN 7-224). This policy will be reflected in the revision of the regulation. The use of fixtures and electrical conduits having an aluminized coated surface should not present a hazard; however, these surfaces should not be repainted with aluminum paint.
File MCHD-SE, Fifth Indorsement, dated 16 August 1963.
From AEC Safety Division to MUCON, Dover, New Jersey.
Longhorn Central File No. 30-1/201.2.

ALUMINUM: REACTION WITH CHLORINATED HYDROCARBONS--ALUMINUM METHYL

- 11:13 (1) Reaction of aluminum with chlorinated hydrocarbons can form explosive compounds, as aluminum methyl, etc.
Letter from Bureau of Mines, 10 June 1958.
Safety Section File, Longhorn Division, 341-11/206.1.
- 11:13 (2) Triethyl-aluminum and Triisobutyl-aluminum: Bulletin by Texas Alkyls, Inc., Houston, Texas. Properties, hazards and safety information.

CONTINUUM: CONTACT WITH TEFLON OR OTHER MATERIALS

- 11:14 (1) Contact between Teflon and aluminum mating parts may create hazardous conditions when propellants are involved. It has been known that mixtures of Teflon and aluminum will burn and can sometimes be set off by friction. Steel tubes, coated with Teflon, emit a smoke when screwed rapidly into aluminum mandrels.
- "Guidelines for Operation", Ordnance Ammunition Command, June 1957.

ALUMINUM: REACTION WITH WATER AND EVOLUTION OF H₂

- 11:21 (1) Gassing (evolution of H₂) results if water is introduced into TPH-8041 (Pershing) propellants.
Trip Report to Huntsville Division of Thiokol, 9-11 January 1961, Joe Murphey, Longhorn Division of Thiokol.
Also See Letter to J. A. Farr, Jr., 11 December 1958, from Thiokol, Huntsville Division.
- 11:21 (2) Gas samples were taken from swollen TRX-135D propellant dog bones. The propellant was water soaked and swelling indicated the evolution of gas. Analysis showed 60% - 70% of the gas to be hydrogen.
Thiokol, Longhorn, Laboratory Report 13 January 1961. File 332-10.

ALUMINUM: INCREASED/DECREASED SENSITIVITY IN CERTAIN PROPELLANTS

- 11:23 (1) It appears the presence of aluminum in PBAA propellants does not increase or decrease the hazard potential of the propellant. Therefore, aluminum is considered neither as an oxidizer nor as a fuel in volumetric ratios ranging from 2.1 to 3.87.
Thiokol Report No. 43-59, Reference 5, Page 3, Container 108, Longhorn.
- 11:23 (2) Sensitivity test on 2056D experimental mix (same as 2026 series except that the alcohol is added to the mix after the addition of AP and resorcinol; ordinarily alcohol is added at the beginning of the mix) showed samples containing aluminum were less sensitive than the previous series which contained no aluminum.
Reference 2.
- 11:23 (3) Tests were conducted to determine the hazard potential of composite solid propellants of the PBAA propellant family. Formulations containing from 65% to 86.8% oxidizer and 0% to 20% aluminum were tested. No evidence of sustained detonation throughout the propellant mass was noted during these tests. Although some formulations showed slight participation in the area adjacent to the initiator, this participation did not propagate through the propellant. From these tests, it was concluded that the presence of aluminum does not increase or decrease the hazard potential of the propellant.
Reference No. 5.
- 11:23 (4) See 25:11 (1) where aluminum increases the hazard in liquid-carrier "quick-mix" process of solid propellant manufacture when the slurry becomes partly dried out.

ALUMINUM: COMBUSTION CHARACTERISTICS IN PROPELLANTS

- 11:24 (1) "Study of Combustion Characteristics of Aluminized Solid Rocket Propellants", by B. C. Harbert and J. A. Keller, Phillips Petroleum Company.
This report is the result of a study to: 1) learn more about combustion of aluminum during the burning of propellants, 2) develop a research method for measuring the effect of additives on the combustion of aluminum in propellants, and 3) find methods of modifying combustion behavior of aluminum in castable propellants, with the ultimate goal of improving engine performance of these propellants.
Reference No. 9, Volume IV, Page 211.
- 11:24 (2) See 13:11 (8).

AMMONIUM PERCHLORATE: CHEMICAL, PHYSICAL, AND STABILITY TESTS

- 12:11 (1) Description of certain testing devices for testing sensitivity of ammonium perchlorate and other material is described, such as impact sensitivity devices developed by Jet Propulsion Laboratory, auto ignition devices by Rocketdyne, friction sensitivity apparatus by Rocketdyne, spark ignition, and subsurface ignition devices.
Rocketdyne R-4069, "Report of Special Explosivity Tests", 14 October 1960. Longhorn Container 108, Safety and Security Department.
- 12:11 (2) Decomposition of Ammonium Perchlorate: Studies on the effects of additives on the thermal decomposition behavior of NH_4ClO_4 were made by performing differential thermal analyses on dry mixtures of 99% NH_4ClO_4 and 1% additives. The DTA results have revealed that additives can affect the thermal decomposition behavior of NH_4ClO_4 in any one of four distinct ways. Reference No. 3, page 211.
- 12:11 (3) Impact Sensitivity of high purity ammonium perchlorate is comparable to that of most high explosives. Requires the equivalent of 10 grams of mercury fulminate or an XC-32 Detonator to detonate the dry material. Very insensitive as explosives go, but more sensitive than the sodium, potassium, and magnesium compounds; drop test, minimum: 86 in. for 11 lb. weight. Insensitive to severe friction test. Heating small quantities does not cause explosion; temperature of explosion = 1308°C ; heat liberated = 344.5 cal/kg . Ammonium perchlorate is thermally stable below 300°F . Decomposition begins at 130°C , becomes explosive at 360°C . Metallic contamination increases the sensitivity of ammonium perchlorate; certain metallic perchlorates are extremely sensitive. The handling of solution and wet solid NH_4ClO_4 is not hazardous, but the drying process presents a fire and possibly an explosive hazard. NAVORD Report 7147, (Vol. 1), "Chlorates and Perchlorates, Their Manufacture, Properties, and Uses".
- 12:11 (4) Detonability of Ammonium Perchlorate: In redesigning their continuous mixing facilities, Clin Mathieson ran some tests of ammonium perchlorate to determine the hazard of material transfer between ingredient feeder (AP) and the continuous mixer. Using 2 and 3-inch diameter by 4 ft. cardboard tubes, loaded loosely or packed with up to 40 lbs. of AP, they placed as many as four tubes end-to-end in a horizontal position. Variations in tube arrangements included use of 90° bends. Detonation of the AP was accomplished through the initiation of booster charges of 100 and 200 grams with a No. 6 blasting cap. Conclusions drawn from these tests show: 1) The critical diameter of tubes confining a static bed of 40 microns of ammonium perchlorate is something less than 2 inches. Critical diameter is defined here as that diameter which must be employed to give a reasonable guarantee that a detonation in one end of the stream will not propagate from the source through the bed of material. In all tests with 3-inch tubes, detonations were established and propagated the full 200-inch length. Some detonations were established in 2-inch tubes; propagation in this case, however, ceased at about 100 inches. 2) No reliance can be placed on 90° bends stopping a detonation in conduits carrying ammonium perchlorate. In each case, the detonation wave transferred around the bends and traveled on down the tubes. This may be due to the low detonation rate as compared to high explosive materials. 3) If critical

diameters are exceeded, no reliance can be placed on employing open or slotted rather than closed tubes. 4) When critical diameters are exceeded, a detonation will travel the full length of the tube, regardless of its length. 5) A light blowout material is ineffective in halting propagation of a detonation.

Mr. James C. Barr, Manager, Development and Engineering Branch, Olin Mathieson. Reference No. 7, pages 3 - 4.

- 12:11 (5) Summary of IRECO Report, "Explosive Properties of Ammonium Perchlorate", Table 1. Experimental results and recommendations are given. Experimental results include such data as: mean impact sensitivity, minimum booster, critical diameter, detonation velocity, high velocity impact test, detonation velocity, high velocity impact test, detonation pressure, card gap sensitivity test, and sympathetic detonation test.
Letter from H. W. Ritchey, Vice President, Rocket Operations, Utah Division.
- 12:11 (6) The Mechanism of Deflagration of Pure Ammonium Perchlorate, by Raymond Friedman and Joseph B. Levy, Atlantic Research Corporation.
Results of new deflagration experiments with ammonium perchlorate, both pure and catalyzed, are given. These include studies of rates of deflagration, surface temperature, and effect of incident radiation on deflagration. Ammonium perchlorate will not normally deflagrate below 22 atmospheres. However, a radiant-heating apparatus (shown in Figure 4, page 366) was found to produce conditions permitting steady deflagration at atmospheric pressure. (The radiant-heat source is a 1000-watt lamp.) Reference No. 9, Vol. IV, page 351.
- 12:11 (7) Impact Sensitivity becomes critical in cases where the sludge (AP in hexa-methyldisiloxane) is not completely wetted by the carrier (a fact substantiated by analogous test with AP and kerosene reported by the Bureau of Mines, Reference 2). Their attempts to initiate detonation were successful only when there were voids in the sludge caused by incomplete wetting or entrapped gas or vapor.
Rocketdyne "Report of Special Explosive Tests", page 30.
Container 108, Longhorn.
- 12:11 (8) No Ignition of Dust Clouds of ammonium perchlorate could be obtained in air at normal temperatures and pressures, either by high voltage electrical induction sparks; by electrical condenser discharge sparks; by a (40-volt, a.c., 7.5 ampere) carbon arc; or by the timed flame of a 75 mg. tuft of gun cotton used in the test equipment. Dust clouds of ammonium perchlorate were not ignited during rapid dispersal of small quantities through the laboratory furnace which was heated to various temperatures, ranging up to 750°C. However, dust clouds (1 oz. per cubic foot) at initial temperature of 100°C and one atmospheric pressure, using as ignition source flame from 300 mg. and 500 mg. tufts of gun cotton respectively, were ignited and relatively mild explosions resulted within the bomb test.
Bureau of Mines, Report No. 3705, 5 September 1953, Container 108, Longhorn.

AMMONIUM PERCHLORATE: EXPLOSIVES HAZARDS IN HANDLING AND STORAGE

- 12:12 (1) Detonation of two drums of ammonium perchlorate in a mixer bay is believed to have initiated by high velocity particles rather than by the detonation wave front from a mixer explosion. Olin Mathieson Chemical Corporation. Minutes of T&SC Meeting, 15 February 1960, Safety and Security Department File, Longhorn.
- 12:12 (2) Detonation of ammonium perchlorate is demonstrated by filling an 8" x 38" steel cylinder with a plate welded on one end and initiating with an explosive charge. Huntsville Division, February 1960. Minutes of T&SC, February 1960, Safety and Security Dept. File, Longhorn.
- 12:12 (3) The bulk density of ammonium perchlorate explosives has a considerable effect on the sensitivity of detonation. (1909) Reference No. 1, page 136.
- 12:12 (4) Ammonium Perchlorate Storage Hazard: A study to evaluate the hazards connected with ammonium perchlorate storage and handling was conducted by the Utah Division of Thiokol Chemical Corporation and International Research and Engineering Company, Inc. This study shows that the critical detonation diameter of powdered ammonium perchlorate is in the order of 5" to 6", and that the detonation velocities approaching 4000 meters per second are obtainable in diameters of 2' and above. As a result of these studies, eight recommendations for handling ammonium perchlorate were made. These recommendations establish higher safety standards for AP storage than are now in use. Letter, dated 24 March 1961, from H. W. Ritchey, Vice President, Rocket Operations, Thiokol Chemical Corporation.

AMMONIUM PERCHLORATE: SENSITIVITY INCREASED/DECREASED BY IMPURITIES

- 12:13 (1) Decomposition of ammonium perchlorate is fairly rapid @ 220°C, still more rapid at 240°C, but negligible at 210°C. Fe_2O_3 has a negative catalytic effect on the first decomposition step and some positive effect on the second step.
Reference 1, page 207.
- 12:13 (2) MnO_2 increases the resistance of ammonium perchlorate to shock.
Reference 1, 1910, page 137.
- 12:13 (3) Traces of NH_3 salts and chlorates in NH_4ClO_4 are to be avoided due to formation of sensitive NH_4ClO_3 .
- 12:13 (4) The sensitivity of perchlorate explosives may be increased by small amounts of impurities; for example, a trace of cupric chloride, or .005 to 1.0% of an alkali or alkaline earth chlorate, will greatly diminish the resistance of NH_4ClO_4 to shock. This is believed to be due to the formation of NH_4ClO_3 , a very unstable compound.
Reference 1, pages 135 - 136; also Reference 6, page 41.
- 12:13 (5) Ammonium perchlorate was found to be most sensitive to ignition at a moisture content of 0.02% to 0.5%, particularly when it is contaminated with copper and possibly in the presence of other metallic contaminants.
Reference 1, page 215.
- 12:13 (6) Additional safety precautions taken at the Western Electrochemical Company's plant included rapid drying through the critical range of moisture content (0.02% - 0.5%).
Reference 1, page 216.
- 12:13 (7) Copper chromite increases the ignition susceptibility of ammonium perchlorate dust. Ammonium perchlorate - copper chromite dust mixtures in air, ammonia - air, or ammonia - nitrogen are ignitable by spark ignition. A gas explosion above a propellant mix can readily be propagated to the propellant if oxidizer dust is present. These phenomena do not occur if an inert atmosphere such as nitrogen is maintained above the mix.
Reference 3, page 37.
- 12:13 (8) Coated Ammonium Perchlorate: Ammonium perchlorate coated with 0.2% tri-calcium phosphate, and containing no organic fuel impurity, can be detonated. We have good evidence to support our belief that a fuel impurity is not necessary.
Mr. James Barr, Manager Development and Engineering, Olin Mathieson.
Reference 7, page 5.

AMMONIUM PERCHLORATE IN EXPLOSIVES AND PROPELLANTS

- 12:14 (1) Maximum Brisance: Variation in the nature of the combustible ingredients have a very marked effect on the optimum charge density required for maximum brisance. Some mixtures require high compression; in others, maximum brisance is obtained when the charge is entirely uncompressed. At low loading densities, the proportion of organic material to oxidizer may vary over a wide range without any appreciable effect on brisance, whereas at higher densities the brisance is decreased by increasing the organic material to a point where a deficiency of oxygen results. Reference 1, page 136.
- 12:14 (2) Oxygen Balance: According to two investigators, the "oxygen balance" of ammonium perchlorate explosives should be between 4% and 6%. Reference 1, page 136.
- 12:14 (3) Critical Confinement: In the past we have discussed critical depth and critical pressure as regards to transition from deflagration to detonation. A restatement of this may be in order. It is, in essence, a combination of these two, and maybe we can call it critical confinement, meaning that a fire is underneath the surface of the propellant, and the physical mass inertia of the material is such that it contains this fire until a given deflagration transposes to detonation. T&SC Minutes, 15 February 1960, Longhorn.
- 12:14 (4) Tests by OCO and Thiokol on PBAA propellants indicate that there was no propagation of detonation in any of the samples, but there was some participation in one special formulation containing 100% ground oxidizer. This indicates that particle size, as well as the percent oxidizer, must be considered when determining the hazard classification of propellants. However, this problem is not serious, since formulations containing ground fractions great enough to cause extensive participation of the propellant in detonations cannot be successfully processed. Thiokol Report No. 43-59, "Special Report Detonation Characteristics of PBAA Propellants". Longhorn Container No. 108.
- 12:14 (5) A very small amount of ammonium perchlorate (2½%) was sufficient to bring the sensitivity figure (of a NG/NC base plus acetone plus resorcinol plus alcohol) down from above 600 mm to 200 mm (5 kilo wt., or .02 gr. sample). Below the 50% level, the positive sliding rod test (10 lb. cylinder or "Torpedo" 0.1 gr. sample) gave only crackles and burnings, while at 50% and above the positive tests were sharp explosions or detonations. Reference 2, Container No. 108, Longhorn.

MOISTURE CONTENT

- 12:15 (1) Ammonium perchlorate was found to be most sensitive to ignition at a moisture content of .02% - .5%.
Reference 1, page 215.
- 12:15 (2) Use of desiccants in tote bins is essential, according to test on Aerojet-type oxidizer at Aberdeen, Mississippi. Memo by Process Development, 19 April 1960, File 80/C, Container No. 108, Longhorn.

SENSITIVE MIXTURES WITH SOLVENTS

12:16 (1) Ammonium perchlorate, when acetone wet in the range of 10% to 20% by weight, becomes extremely sensitive to impact and friction.
Reference 2.

STORAGE, SURVEILLANCE, CONTAINERS

12:17 (1) Aluminum Containers for Perchlorates: Mr. V. E. Haninger, ICC, "There's nobody that has appealed to the ICC to ship perchlorate in aluminum; as far as I know the Bureau of Explosives has never been approached on the problem. I don't think we would do it without some sort of a liner, if we do it at all". Mr. R. P. Rice, American Potash and Chemical Company, "We're the largest manufacturer of AP and have done some work with rocket manufacturers on shipping in bulk containers, namely, nestabin, sealbin; some evaluation work has been done on your Kaiser bin. We have ICC approval to ship in aluminum totebins and inverta bins". Dr. Ball, Hercules Powder Company, "We had an incident where an aluminum container got hot enough in a general fire to melt and get involved in this ammonium nitrate; the results were not pleasant".
Reference 7, page 180. Longhorn Safety & Security File 340-1/304.4.4.

ORGANIC SALTS OF PERCHLORATES

- 12:21 (1) The diazonium perchlorate of p-phenylenediamine was reported in 1910 to be the most explosive substance known.
Reference 1, page 66.
- 12:21 (2) Amine Perchlorates: Perchlorates of a great number of aromatic amine will either volatilize or decompose when heated slowly. However, if they are subjected suddenly to high temperature they will explode.
Reference 1, pages 64 - 65.
- 12:21 (3) Methyl, ethyl, and propyl perchlorates are very sensitive to heat, shock, and friction and are extraordinarily explosive.
Reference 1, pages 67 and 214.
- 12:21 (4) Trichloromethyl perchlorate forms perchloric acid with water; it explodes when heated and also at room temperature when in contact with alcohol or other organic substances.
Reference 1, page 214.
- 12:21 (5) Organic Perchlorate: Incident when handling. See 13:11 (10).
- 12:21 (6) Hydrazine Perchlorate fire in September 1963, Wasatch Division.

INORGANIC SALTS OF PERCHLORATES

- 12:22 (1) Lead or Silver Perchlorate: Various complexes of silver or lead perchlorate with organic compounds are liable to explode.
Reference 1, page 209.
- 12:22 (2) Fluorine perchlorate has been found to be particularly unstable.
Reference 1.
- 12:22 (3) Lithium Perchlorate: (LiClO_4), Thiokol, Huntsville Division, 7 Feb. 1958.
While undergoing aging studies in a 250°F oven, a sample ignited and blew open the door.

FUELS: PBAA, LP, MAPO, EXOTIC FUELS, RADIOACTIVE MATERIAL, METALS

- 13:11 (1) MAPO and ammonium perchlorate are not compatible. These two materials when mixed together undergo spontaneous combustion.
Trip Report, Huntsville Division, Thiokol, January 9-11, 1961, Joe Murphey.
- 13:11 (2) Addition of energetic additives as light metal hydrides in binder/oxidizer systems: Mention is made of the work being done by various companies on different additives, e.g., stabilizing coatings for certain hydrides, desensitizing LiAlH_4 , etc., and encapsulating. Sixteenth JANAF Solid Propellant Group bulletin is referred to for greater details.
Mr. Barr, Olin Mathieson Chemical Corporation.
Reference 7, pages 5 - 9.
- 13:11 (3) High Energy Fuel: Papers deal with the following fuels:
- (a) Vinyl-aluminum Compounds and Aluminum Hydride Complexes of Tetrazoles and Hydrazines. Page 1, Vol. III, Reference 8.
 - (b) Light Metal Hydrides and Their Vinyl, Ethyl, and Methylene Derivatives. Page 9, Vol. III, Reference 8.
 - (c) Derivatives of Boron and Aluminum Hydride. Page 23, Vol. III, Ref. 8.
 - (d) Hydrazine Mono and Diborane. Page 35, Vol. III, Reference No. 8.
 - (e) Synthesis and Formulation of BN Components of Solid Propellants. Page 49, Vol. III, Reference No. 8.
 - (f) The Preparation of Aluminum Hydride. Page 71, Vol. III, Ref. No. 8.
- 13:11 (4) High Energy Solid Propellants Containing Encapsulated Materials.
Vol. IV, Page 23, Reference No. 8. (Aerjet-General Corporation).
- 13:11 (5) High Energy Hydride Solid Propellants. (Hughes Tool Company).
Volume IV, Page 65, Reference No. 8.
- 13:11 (6) Research and Development for Carborane Propellants.
Thiokol - Vol. IV, Page 87, Reference No. 8.
- 13:11 (7) High Polymer, Hydrazine Based Solid Propellants. (Bell Aircraft Corp.).
Vol. IV, Page 155, Reference No. 8.
- 13:11 (8) The Effect of Metal Additives on the Combustion and Performance of Solid Propellants. This paper is a general review of the effect of metal additives (to increase the specific impulse and density) on propellants. Experimental technique, photograph studies, combustion characteristics, effect on unstable burning, ignitibility, sensitivity, and detonability are discussed.
Jet Propulsion Laboratory. Reference No. 9, Vol. IV, page 1.
- 13:11 (9) Chemical Modifications of Polyurethane Propellants. Several references also given.
Jet Propulsion Laboratory. Reference No. 8, Vol. V, page 71.
- 13:11 (10) Reaction Motors, 13 June 1961 (Division of Thiokol Chemical Corporation).
Two cubic inch mixer exploded as operator was adding MAPO to experimental organic perchlorate propellant. Operator lost some fingers, sustained other injuries.
- 13:11 (11) MAPO - NH_4ClO_4 Capability Studies by P. O. Blackwell, Thiokol Chemical Corporation. TM-139, Control No. U-A-61-3204.
- 13:11 (12) MAPO - NH_4ClO_4 Compatibility Test. See Technical Note 11-63. May 13, 1963, by G. E. Dolan.

FUMES (Continued)

13:11 (13) See 10:11 (24) MAPO/Ammonium Perchlorate Sensitivity.

DOUBLE BASE PROPELLANTS

13:12 (1) Processing of "Fluid Ball" Composite Base Propellant, by R. E. Coffee, C. R. Culverhouse, and R. C. Cook. Olin Mathieson Chemical Corporation. Reference No. 8, Vol. IV, page 255.

PYROTECHNICS

13:13 (1) Mr. Milton Cutler, U. S. Army Chemical Warfare Laboratories. Pyrotechnics of propellants and fuels. Drying, mixing and blending, dust, bulk, etc. Reference 6, pages 24 - 28, Thiokol Longhorn Safety & Security Dept. File.

BURNING RATE CATALYST

- 14:10 (1) The effect of metal additives on the combustion and performance of solid propellants is summarized in this paper. Combustion characteristics of many metals used in propellants, whether they be used for ballistic modifiers or performance increase, is given. Compatibility of the additive in the sense of chemical stability of the resulting composition is discussed; for instance, aluminum used in polysulfide-ammonium perchlorate type propellant is limited in the sense that compositions containing more than 8 wt% produce excessive gasification during cure. Effect of metal additives on shock sensitivity, ignitibility, and detonability are areas in which effects are not clearly understood and defined at present. (Jet Propulsion Laboratory), Reference No. 9, Vol. IV, Page 1.
- 14:10 (2) Burning rate modification of double-base propellants. A. M. Jacobs and J. H. Godsey, Hercules Powder Company. Paper deals with lead compounds used as burning rate modifiers. Reference No. 9, Vol. IV, Page 41.
- 14:10 (3) Modification of Burning Characteristics of Solid Propellants. Frank J. Kushibab, Thiokol Chemical Corporation. Alteration of the burning rates of solid propellants is being studied by two general techniques. The first method is the incorporation of selected additives (pyromellitic acid, cobalt, etc.) into the propellant compositions. The second method involves the study of the correlation of chemical and molecular structure to burning rate in liquid polymer-ammonium perchlorate system. Results obtained from these two approaches to the problem are summarized. Reference No. 9, Vol. IV, Page 79.
- 14:10 (4) Factors Governing Burning Characteristics of Composite Solid Propellants. Atlantic Research Corporation by R. Friedman, J. B. Levy et al. Factors which govern the burning characteristics of composite solid propellants are discussed. The results of both experimental and theoretical investigations are used as a basis for conclusions and hypotheses as to the mechanism of combustion. Included in this discussion are the effects of burning rate on pressure, particle size of inorganic constituents, catalysts, and heat transfer by means of metallic wire. Reference No. 9, Vol. IV, Page 97.
- 14:10 (5) Plastic Propellants: Compositions of Improved Performance Covering a Wide Range of Burning Rates, by B. H. Newman et al. Explosives Research and Development Establishment, Waltham Abbey, Essex, England. By variation of oxidant particle size, and by choice of catalyst, in the basic formulation ammonium perchlorate (82-89%)/polyisobutene (9-15%)/surface active agent (1%/catalyst (0-2%)), burning rates in the range of 1.67 to 0.67 inch/s. (at 1000 p.s.i.a.) have been obtained. Of the catalyst studied, copper chromate was outstanding for increased burning rate, and titanium dioxide was the most effective resonance suppressor. For burning rates in the range of 0.6 to 0.15 inch/s. (at 1000 p.s.i.a.), some of the ammonium perchlorate is replaced by ammonium picrate. Other catalysts are discussed. Reference No. 9, Vol. IV, Page 127.

BURNING RATE CATALYST (Continued)

- 14:10 (6) Extension of Burning Rate of Composite Propellants (utilizing a polybutadiene-acrylic acid "PBAA" type copolymer as the binder) of the rubber base type. Olin Mathieson Chemical Corporation. Some investigations were also made on propellants containing binders consisting of a polybutadiene-styrene-acrylic acid terpolymer, modified polyisobutylene or polyurethane prepolymer. Other studies concerned effect on burning rate and pressure exponent of amount and particle size of the ammonium perchlorate, use of aluminum and several inorganic boron materials as energizers, and special additives for burning rate enhancement or depression.
- 14:10 (7) Burning Rate Modification Studies. Rohm & Haas Company. This paper deals with the dekene and dekasene-type decaborane derivatives. Increased burning rates result when added to nitrate ester-type propellant. When dekene derivatives were used as the sole binder with ammonium perchlorate, very fast and reproducible burning occurred (3.0 in/sec. at 1000 psi). Reference No. 9, Vol. IV, Page 177.
- 14:10 (8) Burning Rate Modification of Castable Propellants Comprising Ammonium Perchlorate and a Polybutadiene Binder. J. P. Blackwell, Phillips Petroleum Company. Reference No. 9, Vol. IV, Page 193.
- 14:10 (9) Suppression of Combustion Instability by Chemical Additives, by H. Cheung and R. L. Lou, Aerojet-General Corporation. Reference No. 9, Vol. IV, Page 233.

PARTICULAR INCIDENTS

- 14:11 (1) Hydrazonium Nitroformate - (high energy oxidizer). Explosion occurred while reducing size in a motor and pestle. Huntsville, Alabama. C. No. C-A-60-6887. Container 106, Longhorn Safety and Security Department.
- 14:11 (2) Impact Sensitivity of Some Perchlorate Propellants: Table shows the impact sensitivity of various formulations with lithium perchlorate, ammonium perchlorate boron, aluminum and copper chromite. Reference No. 3, page 76.
- 14:11 (3) Lithium Ammonium Hydride: Spontaneous fire occurred in a waste barrel behind the Chemical Laboratory, Huntsville. See Accident Reports and Abstracts, Thiokol-Huntsville, C. No. 60-09490.
- 14:11 (4) See 10:12 (20). Propellant cure incident due to presence of ammonium dichromate in the propellant formulation. Reference No. 12, page 58.

HIGH ENERGY OXIDIZERS

15:10 (1) Symposium on High Energy Oxidizers: Papers deal with the following high energy oxidizers:

- (a) Direct Fluorination of Organic and Inorganic Compounds. Vol. III, page 81, Reference No. 8.
- (b) Oxidizer Synthesis. Vol. III, page 91, Reference No. 8.
- (c) Fluorine Containing Oxidizers. Vol. III, page 103, Reference No. 8.
- (d) The Direct Fluorination of Nitrogen Compounds. Vol. XII, page 113, Reference No. 8.
- (e) Heats of Formation of Some Binary Fluorides of the Light Elements. Vol. III, page 125, Reference No. 8.
- (f) Physical and Chemical Properties of Nitronium Perchlorate. Vol. III, page 139, Reference No. 8.
- (g) The Thermal Decomposition of Nitronium Perchlorate. Vol. III, page 169, Reference No. 8.
- (h) Hydrazine Perchlorate Oxidizer for Solid Composite Propellants. Vol. III, page 173, Reference No. 8.
- (i) Metal Perchlorate Coordination Compounds. Vol. III, page 187, Reference No. 8.
- (j) Synthesis of New Inorganic Ozonides. Vol. III, page 195, Reference No. 8.

15:10 (2) See 10:13 (5), N_2F_4 Oxidizer Incident.

15:10 (3) See 10:13 (6), HP_2 Oxidizer Incident.

15:10 (4) See 10:16 (9), BeH_2 /High Energy Fuel Laboratory Fire.

PROCESS HAZARDS: SCREENING, MIXING, FINISHING, ETC.

SCREENING AND GRINDING OXIDIZER, CURING AGENTS, ETC.

- 20:10 (1) Grinding Ammonium Perchlorate: Thiokol, Huntsville Division, 12 June 1958. Fire occurred when feeding oxidizer to a micro-pulverizer. Bolt head, apparently rusted by oxidizer, had fallen into pulverizer.
- 20:10 (2) Grinding Ammonium Perchlorate: Thiokol, Huntsville Division, 4 December 1959. A fire occurred in a micro pulverizer during the grinding operation. Cause: Undetermined. Automatic deluge system extinguished the fire. A hopper screen was not used at the time.
- 20:10 (3) Grinding Ammonium Perchlorate: Thiokol, Huntsville Division, 26 June 1959. During oxidizer grinding operation, a V-belt transmission drive on the feed mechanism overheated and burned. The AP in the hopper or the ground AP in a container did not burn. Approximate Cause: Friction on V-belt due to misalignment or slippage.
- 20:10 (4) Explosion of Electric Motor in Oxidizer Processing Cell: 8 September 1949, Aerojet Engineering Corporation, Azusa, California. One fatal injury. Extensive building damage. The explosion occurred in a 1 HP electric motor (approved for hazardous locations) into which potassium perchlorate had infiltrated and become contaminated with lubricant in the windings. This explosive mixture was ignited upon starting the motor or shortly thereafter by unprotected attendant. The motor was used to drive an ice crusher into which perchlorate is first charged for crushing. (Grinding operations are performed with personnel at separate, barricaded locations.) Silicone grease was being used in bearings of oxidizer processing equipment. Since it was demonstrated in this explosion that perchlorate dust infiltrates a sealed electric motor over a period of years, taking of tachometer readings at oxidizer grinding equipment, even with the hopper uncharged, may present a personnel hazard.
Reference 12, page 54.

PNEUMATIC CONVEYING, SYNTRON FEEDER

- 20:11 (1) Discussion following a paper given by Mr. C. James Barr, Manager, Olin Mathieson Chemical Corporation, on "Detonability of Ammonium Perchlorate and Propagation of the Detonation". Mr. Ball of Hercules Powder Company, asked if any work had been done on thin layers of AP as you get on a Syntron feeder. The answer was, "No, tests were on thicker layers". (Tests showed that no reliance can be placed on 90° bends stopping a detonation in conduits carrying AP in 3" tubes 200" long; the critical diameter is exceeded, apparently, as in 2" tubes the detonation ceased at about 100".) Dr. Ball pointed out in transferring AP, a thin layer, as you would have in using a Syntron feeder, may prevent propagation, whereas a screw or something else would not. For test details by Olin Mathieson, see 12:11 (4).
Reference No. 7, page 5.
- 20:11 (2) Evaluation of Processing Hazard and Pneumatic Conveying of Hazardous Material: It is claimed handling cost and less personnel exposure can result if pneumatic conveying is utilized. Two principal precautions are necessary: static and foreign material. Static accumulations on propellant (smokeless powder) being conveyed can be dissipated by control of the humidity. Investigations have proved that if a certain minimum differential is maintained between the velocity at which the air is moved through the system and the velocity at which the propellant moves through the system, the flame of any pre-ignited granules of propellant will be "snuffed out" upon entering the system; and it is not possible to propagate a combustion throughout the system. Studies with a flame propagation with a pneumatic conveying system revealed the ignition of a propellant stream is not possible with the flame from a 2000°F oxyacetylene welding torch when a minimum air stream velocity of 6000 f.p.m. is maintained.
Mr. J. E. Settles, Hercules Powder Company, Reference No. 7, pages 74-81.

HEALTH HAZARDS

- 20:12 (1) Outline of the medical and safety steps to be taken in handling materials used in propellants. Toxic properties of nitroglycerine, resinous fuel binders, Styrene monomer, polyurethane, isocyanate, beryllium, diborane, lithium, and aluminum.
Dr. R. N. Duguid, Scientific Director, USAEHL, Army Chemical Center, Md.
Reference No. 7, pages 46 - 53. Longhorn Safety & Security Dept. Container.
- 20:12 (2) Hydrogen Sulfide Evolved When Colloidizing: Thiokol, Wasatch Division, December 1961. When colloidizing LP-205 and sulfur, two men were overcome by hydrogen sulfide gas. Heat generated during colloidization hastens the formation of the gas.
Reference: Memo from Wasatch Division, 6 December 1961.
- 20:12 (3) Evolution of Hydrogen Sulfide Gas after Colloidizing: Thiokol, Huntsville Division. After colloidizing a mixture of LP-205 and sulfur, an operator was stirring the mixture by hand, became dizzy and fell, striking his head against the concrete floor.
Memorandum 24 April 1961.
- 20:12 (4) Teflon: Toxic properties considered not so severe as once thought to be. See Medical News Letter, United States Navy, Vol. 33, No. 2, page 21.
Safety File No. 1/500.
- 20:12 (5) Toxicity of Metal X: November 1963.
A.E.C. Limits: Instantaneous Dose - 25 micrograms per cubic meter.
Average Industrial Plant Dosage - 2 micrograms per cubic meter, 8 hours.
Average Out-of-Plant Dosage - .01 micrograms per cubic meter, 8 hours.

TOOLS, SOLVENTS, DEGREASERS

- 20:13 (1) Aluminum "Punch" Rod: Dope house fire, 6 September 1961.
A flash occurred when an operator "punched" a lump through a mild steel grate with an aluminum tamp. Later tests proved the aluminum tamp (containing 0.25% copper, 0.60% silicon, 1.00% magnesium, and 0.25% chromium) will spark readily when it strikes steel with a glancing blow. It is stated the aluminum is "non-sparking". Recent tests indicate that practically all combinations of metal couples can cause ignition of explosive atmospheres by impact. Alloys are particularly prone to cause ignition. Only pure soft aluminum tips should be used on tamps.
ASESB Explosive Incident Report No. 38.
- 20:13 (2) Trichloroethylene Vapor Degreaser: Thickol, Huntsville Division, 20 March 1958.
A minor explosion occurred in the vapor degreaser at the beginning of the work shift. Approximate Cause: Auto-ignition of the sediment which was settled on and around the exposed coils. Ignition temperature was tested on one sample and found to be 248°F. Analysis of a sample was as follows: Grease, DPG, GMF, and LP - 10.66%; Al. Powder - 14.09%; Thermax - 17.35%; Metal Silver and Unknown - 14.36%; Miscellaneous - 13.25%.
- 20:13 (3) Vapor Degreaser Coils: Thickol, Huntsville Division, 14 January 1958.
A fire was started by an operator chipping the cured sediment from the coils of a degreaser distillery with a screwdriver. The major portion of contaminant was found to be oxidizer and aluminum.
- 20:13 (4) Trichloroethylene vapors can form flammable mixtures with air at atmospheric pressures and at temperatures above about 86°F. This temperature corresponds to a lower limit of flammability in air of about 12.5 volume-percent trichloroethylene (air saturated with trichloroethylene vapors). The higher upper limit is believed to be 90 volume-percent (at 180°F). Reference: Bureau of Mines Investigation Report No. 6190, "Flammability of Trichloroethylene".

MIXING HAZARDS

NH₃ and Other Combustible Gases/Oxidizer Dust, Initiation Over Propellant

- 21:11 (1) Solvent Phase Ignition Test: Indian Head, Maryland.
Experiments revealed that in most instances, a vapor phase ignition over a hybrid formulation (formula containing NG, AP, W6, and other ingredients) will ignite the mix which will burn very rapidly with high heat release. Reference No. 2.
- 21:11 (2) Gas and AP Dust Explosion Studies: Aerojet-General, August 1957.
Oxidizer dust shifts the explosive limits of combustible gases in the direction of lower limits; "safe" gas mixtures in the presence of oxidizer dust become explosive. Tests prove that: 1) an explosive gas-oxidizer dust mixture can be generated over propellant mixes containing HTDA and AP. Analysis revealed that these formulations not only evolve an appreciable amount of ammonia, but also small amounts of low-molecular-weight ketones, aldehydes, oxides, alcohols, and traces of hydrogen; 2) the gas dust mixture can be exploded by an electric spark; and 3) the gas-dust explosion will propagate to the propellant mass. This sequence of events, initiated by an electro-static spark, may have been responsible for the mixer explosion on 10 August 1957. See 10:11 (9), Reference 3, pages 31 - 38.
- 21:11 (3) Testing Devices and Procedures Are Explained. A saturated acetone-ethanol atmosphere instantaneously ignites by a spark. The addition of ammonium perchlorate suspension caused the oxidation to be more vigorous, but no detonation. Ignition of a double base propellant mix (NC and NG) by burning acetone-ethanol-ammonium perchlorate atmosphere is difficult, but ignition of a hybrid mix is very easy. Reference No. 2.
- 21:11 (4) When the air in the explosive gas mixture (over propellant in a mixer) was replaced by nitrogen, the initial gas explosion leading to propellant ignition did not occur. Hence, it appears that an efficient nitrogen sweep over the propellant mix is an effective method of minimizing the gas explosion hazard during mixing. Aerojet-General, Reference 3, page 35.
- 21:11 (5) Gas-phase-initiated explosions occurred with ANP-2622AP* propellant formulated with and without aluminum. Moving pictures obtained with conventional and high-speed cameras showed the sequence of events leading to the explosion. This sequence was: 1) a gas-oxidizer dust explosion, 2) a fire in the oxidizer stream, 3) ignition and explosion of the propellant. The time interval between events and the nature of the explosion differed considerably. The intervals were comparatively long with the propellants containing no aluminum, and the explosion was of low intensity. Tests run in laboratory mixer. Reference 3, page 37.

*A polyurethane binder, an amine cross-linker (monohydroxyethyl-trihydroxypropyl ethylenediamine, i.e., HTDA), ammonium perchlorate, and aluminum.

NH₃ and Other Combustible Gases/Oxidizer Dust, Initiation Over Propellant (Continued)

- 21:11 (6) An oxidizer stream to carry fire into the propellant mass is apparently an essential element in the propagation process. (Exploding of combustible gas mixtures over propellant, while mixing was in progress, did not ignite the propellant; but propellant ignition invariably resulted when both the explosive gas mixture and oxidizer dust were present.)
Reference No. 3, pages 35 and 37.
- 21:11 (7) Tables show the composition of gases evolved from propellants during mixing, their flammability limits, and the explosiveness of dust (oxidizer, copper chromite, carbon black, aluminum, etc.) in various gas atmospheres, including nitrogen.
Reference No. 3, pages 39 - 40.

MIXING HAZARDS (Continued)

Contamination--Chemical, Foreign Objects

- 21:12 (1) Experimental tests were run with a one-pint mixer (Baker Perkins type), to determine what factors are most likely to initiate mixer fires. All tests were made with a propellant containing 85% AP and 15% binder. Metal turnings 0.040 to 0.055 inch wide and grit 0.039-0.078 inches in diameter were added to the mix. No fires or evidence of propellant decomposition occurred during these tests. Phillips Petroleum Company.
Reference No. 3, pages 77 - 78.

Oxidizer Concentration, Settle-Out, Voids, Wetting

- 21:13 (1) See 23:12 (1) for the hazard associated in a liquid-carrier solid propellant mixing process, when ammonium perchlorate and aluminum are permitted to settle-out.
- 21:13 (2) For oxidizer concentrations which change class of propellant, see 31:12.
- 21:13 (3) Voids: Detonating potential of LP-33 fuel/ammonium perchlorate propellants when in a state of high porosity as during processing. Samples tested containing 82% ammonium perchlorate by weight or less, showed no evidence of even partial detonation.
Details of test method given. Huntsville, Alabama, 1955.
Reference No. 3, page 7, Container 108, Safety & Security Dept., Longhorn.
- 21:13 (4) Oxidizer/Fuel Ratio, Hazard Class, Test: Detailed tests are given which prove both cured and uncured TRX-135 propellants, having a 2.24 to 1 volumetric ratio of oxidizer to fuel, only burn; and structural damage would result only when buildings are improperly vented or without reinforced concrete walls. LP propellant.
Reference No. 4, pages 1 and 5, Container 108, S&S Department, Longhorn.
- 21:13 (5) Large tests with 77% oxidizer have not been conducted - OCO. Sufficient tests were conducted to qualify 74% or less oxidizer as Class 2B.
Container 108, Safety and Security Department, Longhorn.
- 21:13 (6) Attempts to cause transition from deflagration to detonation of 500 pounds of uncured propellant (ammonium perchlorate and LP-33 fuel, volume ratio of less than 2.85) contained in a barrel, by initiating with squibs buried in the mass, failed. Negative results were also obtained with 50 grams of tetryl and a No. 8 detonator. However, when two squibs were buried at a depth of 23 inches in a 240-pound mass of propellant, representative of the composition during initial stages of "reverse mixing" (volume ratio greater than 4), detonation occurred. Detonation did not occur, however, when the squibs were buried at a depth of 22 inches.
Huntsville, Alabama, Reference No. 3, page 8.
- 21:13 (7) See 23:10 (1). Test by Thiokol at Huntsville in 1957 showed that detonable mixtures are present in a mixer during the first part of "reverse" mixing. The propellant tested was composed of 88% AP, 2% iron oxide, 1% magnesium oxide, and 9% LP.

Energetic Additives as Light Metal Hydrides, Encapsulated Materials

- 21:14 (1) Energetic Additives in Binder/Oxidizer Systems: Reference is made to the 16th JANAF Solid Propellant Group Meeting in Dallas. Progress made by the various companies is reviewed. Hazards associated with this type propellant are also discussed.
Mr. Barr, Olin Mathieson Chemical Corporation.
Reference No. 7, pages 5 - 9.
- 21:14 (2) The author of this paper claims that their "Fluid Ball" of continuous propellant mixing has succeeded in combining a number of rather energetic and sensitive materials into a castable, case-bondable propellant. Three metering stations feed continuously into a small mixer the casting solution, ammonium perchlorate, and Ball Powder/aluminum blend. One incident occurred in this system. A shock initiated in this little mixer, very probably as a result of an underfeed of the liquid metering station or an overfeed of the perchlorate system. This resulted in an overly dry material in the mixer.
Mr. Barr, Olin Mathieson Chemical Corporation, Reference No. 7, pages 81 - 83.
- 21:14 (3) The Future of Encapsulated Materials Predicted.
Dr. H. Shuey, Rohm and Haas Company.
Reference No. 7, page 189.

MIXING HAZARDS (Continued)

Incidents--Causes, Theory

- 21:15 (1) Mixer Explosion: Indian Head, Maryland, August 1959, Baker Perkins type mixer. Opinion of the board is that the explosion within the body of the mixer was initiated in the left forward gland of the mixer and was caused either by blade deflection, which impacted the blade shaft against the bronze insert, (query, maybe cu-perchlorate complex) which set up a frictional environment causing ignition, or detonation of the material within the gland area. Reference 2.
- 21:15 (2) Same incident in 21:15 (1) above. "...ammonium perchlorate, wet with acetone in the range of 10% to 20%, is most sensitive. Inasmuch as this particular formulation, containing a high percentage of this material, plus acetone, was present, it is felt that it was the most likely source of ignition. If the point of initiation is as presumed, the ignition was communicated to the mass of propellant which detonated due to the confinement of the closed mixer and/or the confinement of the weight of the mass itself." Reference No. 2.
- 21:15 (3) Same incident in 21:15 (1) above. Experience with mixer incidents at this activity have indicated that an explosion will usually not result from an ignition (solvent or propellant), if there is not confinement of the propellant.
- 21:15 (4) "Tucking-in" of Flaming Surface Material: An additional hazard, common to all types of mixers, is the continued movement of the blades following an ignition of the charge. It is considered likely that delayed explosions are due more to the "tucking-in" of flaming surface material than "burning to detonation" and unexplained so-called "hotspots". This is not intended to suggest that burning to detonation does not occur. Phillips Petroleum Company, Reference No. 3, page 17.
- 21:15 (5) Faulty Bearing: In one case (fire in Baker Perkins type mixer) it is believed that the ignition may have been caused by a faulty bearing housing, allowing one of the incorporator blades to come into contact with the pan walls. Explosive Research and Development Establishment, Waltham Abbey. Reference No. 3, page 46.
- An automatic system (not described in this paper) could be used to shut down the mixer at the first occurrence of metal-to-metal contact. Phillips Petroleum Company, Reference No. 3, page 84.
- 21:15 (6) "...some other ignitions have been clearly due to 1) sudden adiabatic compression of air above the propellant, or 2) to overheated bearings in contact with the propellant". No incident described in this paper. Reference 3, Explosive Research and Development Establishment, Waltham Abbey. Query: Possibility of 1) when Ko-Kneader screw moved forward on taking up slack? See 10:11 (24).

MIXING HAZARDS (Continued)

Incidents--Causes, Theory

- 21:15 (7) Although the cause of this fire (in Baker Perkins Mixer) was specifically determined, it is very possible that the friction of the mixing blades on large quantities of ammonium dichromate and oxidizer can produce local hot spots sufficient to decompose the ammonium dichromate and initiate the fire. The incident just described involved propellants consisting of 80% ammonium nitrate, 15% copolymer (binder), 3% ammonium dichromate, plus the remaining 2% of other additives.
Gus S. Economy, Safety Engineer, Hill AFB, Utah, Directorate of Air Munitions, Ogden Air Material Area, generally known as OOAFA.
Reference 6, pages 13 and 14. Container 108, S&S Department, Longhorn.
- 21:15 (8) "One fire occurred while adding solvent-wetted ammonium perchlorate to a mixer. This incident was believed caused by friction of the container on the mixer lid, causing ignition of the solvent vapors, or possibly friction of the container on some spilled material on the mixer lid".
L. E. Greiner, Hercules Powder Company.
Reference 6, page 35, Container 108, Safety & Security Dept., Longhorn.
- 21:15 (9) The incident (Baker Perkins type mixer at Longhorn, TCC) may be described as a confined deflagration, probably initiated by a fire in the gland providing subsurface, high pressure ignition of the propellant mass. At this stage of mixing, the power curve indicated consolidation of the mix, but the volume occupied by the propellant indicates a very large percentage of entrapped air at this stage. High pressure ignition of such a system would furnish an extremely high agitation; rates of pressure rise of 10⁷ psi/sec have been obtained. It is the author's opinion, not shared by some of the group, that the mixer lid afforded extra time of confinement to the burning process and contributed to the disruptive effect.
Reference: Opinions of Outside Investigating Board.
- 21:15 (10) Destruction of Large Refinery Unit by Gaseous Detonation: Standard Oil Company, Whiting, Indiana, 27 August 1955.
Investigators reveal technique to determine source of ignition and sequence of events. Luder lines, Chevron Pattern Orientation and low temperature references are described. Technique used in this investigation has potential worth in analysis of propellant explosion incidents.
Reference: Chem. Eng. Progress, December 1957, Vol. 53, No. 12, page 565.
- 21:15 (11) Fires resulting in detonation...presence of interconnected porosity in a charge under confinement...what apparently happens is that the expotential pressure rise can continue up to pressures approaching detonation pressure. When this happens, the deflagration process goes smoothly into detonation. Alternatively somewhere along the pressure rise, shock wave conditions can originate. This can happen in spite of the fact the container will not withstand pressures anywhere near those we are talking about. The process takes place so fast that a ruptured container cannot get out of the way and it therefore remains a container.
Reference No. 3, page 66.
- 21:15 (12) See Reference 11, page 67.

MIXER CRITERIA: DESIGN FINISH, EQUIPMENT, LIDS, ETC.

- 22:10 (1) Recommended that manufacture of high impulse propellants be confined to those mixers of stainless steel construction with a finish specification of 32 micro-inch or better on the interior of the bowl and on the surface of the blades.
August 1959, Reference 2, Container 108, Longhorn Safety & Security Dept.
- 22:10 (2) Direct driving of mixers was replaced with a hydraulic, variable speed unit or a fluid coupling to prevent high torque application which will cause blade deflection when the mixer is operated under high load conditions.
Reference No. 2, Container 108, Safety & Security Dept. File, Longhorn.
- 22:10 (3) Increased mixing hazard in enclosed mixing systems: "...fires resulting in detonation...the presence of interconnected porosity in a charge under confinement...what apparently happens is that the exponential pressure rise can continue up to pressure approaching detonation pressure. When this happens, the deflagration process goes smoothly into detonation. Alternatively somewhere along the pressure rise, shock wave conditions can originate. This can happen in spite of the fact the container will not withstand pressures anywhere near those which we are talking about. The process takes place so fast that a ruptured container cannot get out of the way and it therefore remains a container.
Reference No. 3, page 66.
- 22:10 (4) Mixer Lids: Experience with mixer incidents at this activity have indicated that an explosion will usually not result from an ignition (solvent or propellant) if there is not confinement of the propellant.
Reference No. 2, Container 108, Thiokol, Longhorn Safety & Security Dept.
- 22:10 (5) Laboratory Mixer: A small laboratory mixer is described. Experience in mixing sensitive components as nitropolymer, ammonium perchlorate, and coated lithium aluminum hydride, has demonstrated its safety.
Mr. L. T. Carleton, Aerojet-General Corporation, Reference No. 7, page 9.
- 22:10 (6) Vertical Mixers: Make, tolerances, size, cleaning methods, modifications made for safety, etc.
Reference: Solid Propellant Mixing Survey, by the Solid Propellant Safety Panel of Aerospace Industries Association of America, Inc., 10 October 1963. Published by Rocketdyne, Solid Propellant Division, McGregor, Texas. Pages 107 - 135.
- 22:10 (7) Armed Services Explosive Safety Board, Incident Report No. 153.
A 2400-pound batch of solid propellant formulation was in the early stages of the mix cycle when flames were seen coming from the mixer. The burning inside the mixer evolved sufficient gas to force open the counterbalanced mixer lid. The force of the deluge splashed most of the burning propellant out of the mixer. Cause: Propellant and oxidizer residue was found in the crevice between the metal floor plate and the mixer wall. The sealing material had cracked, permitting contaminated solvent from the cleaning operations to seep inside, and the metal-to-metal contact from the slight vibration of the mixer ignited the residue. A split was also found in the gasket of the mixer lid. The pressure differential caused by 5 inches of vacuum inside the mixer permitted the propagation of the flame through the split gasket into the mixer batch.

MIXER CRITERIA: AUTOMATIC SHUT-OFF, SAFETY DEVICES

- 22:11 (1) An automatic system could be used to shut down the mixer at the first occurrence of metal-to-metal contact. (No system shown in paper).
Reference No. 3, page 84, Phillips Petroleum Company.

Certain tests were run with a Baker Perkins or Process Test Mixer with 85% ammonium perchlorate, 15% binder, (150 grams total, one pint mixer capacity), metal turnings and grit added to the mix in order to determine what factors are most likely to initiate fires in mixers. Mixing time, processing temperature, and blade clearance were all varied for most severe conditions. It was concluded that: a necessary set of critical conditions involving quantity of propellant, degree of propellant circulation, and frequency of metal-to-metal rubbing must be obtained before a fire can be initiated. It appears likely that during normal operations of internal mixers, occasional metal-to-metal rubbing occurs under high loadings of short duration, but conditions seldom become sufficiently critical to initiate propellant fires. (Comment: They varied the clearance of the mixer blades to cause metal-to-metal rubbing.)
Reference No. 3, pages 77 - 84.

MIXER CRITERIA: ROTATING PARTS, BLADES, ETC.

- 22:13 (1) Sigma-Blade: There are no changes in the physical properties of a metal (mixer blade) as a result of fatigue, i.e., crystallization does not exist as a result of fatigue; instead, damage in the form of cracks will begin at the weakest point and progress to failure.
T&SC Meeting, 15 February 1960. Safety & Security Dept. File, Longhorn.
- 22:13 (2) Mixer Paddle Inspection: Mixer paddle components should be inspected for cracks and other discontinuities, using "ZYGL0" or penetrant dye before installation and at frequent intervals thereafter to insure that nothing of a deteriorative nature has occurred by condition of use. Recommendation by Superintendent of U. S. Naval Gun Factory in regard to investigation of mixer explosion. See 18:11 (1).
Reference No. 2.
- 22:13 (3) Glands and gland shoulders should be removed from all mixers (Baker Perkins) used in the manufacture of high impulse propellants, and should be replaced with synthetic substances such as Teflon or other compatible material. Use of nylon is not recommended, because of incompatibility (NG, NC, and 2-nitro-phenylamine hybrid powders containing AP and an additional stabilizer, MgO and resorcinol).
Reference No. 2. See 13:14 (1), Teflon and Aluminum.
- 22:13 (4) Rotating shaft and gland experiments revealed that continued exposure of hybrid propellant (double base propellant with AP) to frictional environment will not ignite the material within the gland area. (Multi-base propellants normally penetrate into the gland area since the gland is not packed, permitting the propellant to flow through the shaft-gland annulus and collect in catch pans. In case of over-solvated propellant during the solvent removal phase of the mix cycle (blow-down), the propellant becomes dry and hard, thereby disrupting flow. The propellant in the dry state is especially sensitive to impact and friction.
August 1959.
Reference No. 2.
- 22:13 (5) Samples were taken from a bearing of a mixer (located in Bay 3) which had been used to mix hybrid 2056 and was located in a bay adjacent to a mixer which had exploded in Bay 4. The mixer located in Bay 4, which exploded, also had been used to mix hybrid 2056. Therefore, it was assumed that some of the same material found in the bearings of the mixer in Bay 3 could have been in the mixer bearings in Bay 4. The analysis of the sample showed the following:

Nitroglycerine	8.1%	Ammonium perchlorate	26.0%
2-Nitrodiphenylamine	0.8	Nitrocellulose	24.0
Other plasticizers	2.1	Copper	0.01
Aluminum	36.5		

Query: Isn't this bad stuff to be in a bearing?
For sensitivity, see 30:11 (6).
Reference No. 2.

- 22:13 (6) The basic design of sigma blade mixers is considered to present excessive hazards, the danger being the blade hubs and packing glands. It was concluded that the most hazardous zones were the packing glands and the spaces between the blade hubs and bowl ends. The space between the blade hub and bowl is so small it can neither be cleaned effectively nor inspected. More important, material which penetrates into these zones is subject to curing and metal bonding. These conditions may be tolerated in the first mix, but the consequences during subsequent mixes may be serious. As a simple solution of improving the conditions of existing equipment, the hubs of the blades are cut back to allow a felt split ring to be placed in position and held in place with a garter spring. After each mixing operation, the area is cleaned and the felt ring is carefully removed. Inspection will show whether penetration has occurred. If not, new felts are fitted and the mixer is ready for use. Canadian Armament Research. Reference No. 3, pages 20 and 21.
- 22:13 (7) Mixer Blade Deformation: The results show that fires were initiated by metal-to-metal contact between rotating and stationary parts within the mixer. This condition can result from deformation of the blades under heavy load or from improper maintenance. These conclusions are consistent with the findings of investigations of accidental fires and the observed deformation of mixer blades under load. Phillips Petroleum Company. See 21:15 (5) for automatic shut-off on metal-to-metal contact. Reference No. 3, page 73.
- 22:13 (8) As a result of a special study on the sensitivity of rubber base propellants with a small mixer, it was concluded that: It is not possible to initiate propellant fires in a mixer by 1) intensive mixing of propellant, 2) friction between the packing gland material and the rotor shaft, or 3) friction between the propellant and the rotor shaft. Phillips Petroleum Company. Reference No. 3, page 83.
- 22:13 (9) Teflon inserts are now used in sigma blade mixers. An explosion had occurred using (packless gland?) all-round clearance of 30/1000. It is believed some AP had worked between the shaft and the gland, and the blade flexing the shaft impacted against the gland and caused ignition. Extreme galling appeared on the shafts themselves, so we are quite sure that the blade was being distorted to the degree where it would contact the gland. Reference No. 6, pages 38 and 39.

Mr. Max Stuckey, Thielkol Elkton, is of the opinion jute makes the best packing. He stresses that the packing should be soaked in thin polymer 48 hours before use. Reference No. 6, page 39.

MIXER TYPE: BATCH MIXING, FORWARD AND REVERSE

23:10 (1) Reverse Mixing: Propellant taken from a mixer during an intermediate stage of "reverse" mixing was used to perform a test to determine if the propellant at this stage is detonable. The mixture contained approximately 88% perchlorate, 2% iron oxide, 1% magnesium oxide, and 7% LP. Four drums of the propellant were tested by initiating with squibs located at the bottom of the drums. The following results were found:

<u>Test No.</u>	<u>Depth Propellant</u>	<u>Weight Propellant</u>	
1	22"	200#	Mild pop.
2	28"	240#	Propellant tamped - High order.
3	28"	240#	Propellant loose - High order.
4	28"	240#	High order, second can 2" from first can also detonated.

Thiokol, Huntsville Division, 6 September 1957 (57-22004).
Longhorn Container 108, Safety and Security Department.

MIXER TYPE: CONTINUOUS MIXING

- 23:11 (1) See 22:10 (3) for: Increased mixing hazards in enclosed mixing systems. Reference No. 3, page 66.
- 23:11 (2) Continuous Mixing: "Fluid Ball" continuous propellant mixing process. Reference No. 7, pages 81 - 83. Also see Reference No. 8, Vol. IV, page 255, Olin Mathieson Corporation.
- 23:11 (3) Continuous Mixing: Problems encountered in continuous mixing solid propellants. Mr. J. R. Crawford, Thicol Chemical Corporation, Longhorn. Reference No. 7, page 83.
- 23:11 (4) Continuous Mixing: Hazard of overly dry material in the mixer. The incident with Olin Mathieson's "Fluid Ball" continuous mixer was believed to be due to an under-feed of the liquid metering station, or an over-feed of the perchlorate system, which resulted in an overly dry material in the mixer. Reference No. 7, page 83.

To prevent a build-up of an overly dry (unbalanced) state in the mixer screw, consideration should be given to a procedure whereby the system could be purged with fuel whenever there is indication that there is trouble in the screw, such as a build-up of perchlorate or other dry ingredients. (Dr. Ball, Hercules Powder Company)
Reference No. 7, page 90.

- 23:11 (5) Progress Report on Continuous Mixing of Castable Propellant at Aerojet General Corporation: This paper deals with the development work and construction of a production facility to insure that the production of the Polaris Propulsion System will combine highest quality and maximum safety with the lowest cost of manufacture. Pilot plant studies were made in the following areas: 1) New Propellant Formulations, 2) Rubber Base Binder, 3) Polyurethane Binder System, 4) Equipment Evaluation, 5) Mixing Studies, 6) Metering Studies, 7) Propellant Pumping Studies, 8) Scale-Up to Production Equipment. A brief description is shown of their full scale production facility with photographs of the mixing equipment. A. S. Cohen, W. Drobot, et al, Process Engineering Department, Aerojet General.
Reference No. 8, page 175, Vol. IV.
- 23:11 (6) New Quality Control Techniques for Continuous Propellant Processing. A successful, continuous propellant stream analysis method, for a 1,000,000 pound per month continuous mixer is described. The instrument measured ammonium perchlorate content, density, and viscosity by a non-destructive method. Photographs, charts, data, and information on the instrumentation is given. Aerojet General Corporation.

NOTE: 21:14 (?), where it is believed that an explosion of a continuous mixer was due to overly dry mix, due either to an under-feed of the fuel or an over-feed of the ammonium perchlorate.
Reference No. 8, Vol. IV, page 275.

MIXER TYPE: LIQUID CARRIER, "QUICKMIX" PROCESS

- 23:12 (1) General Reference: Rocketdyne, McGregor, Texas. R-4069.
Report on Special Explosive Test - A report of results and tests on their liquid-carrier (N-Hexane, N-Heptane, 2-Propanol or HMDS - Hexamethyldisulfoxane) solid propellant continuous processing system, designated as "Quickmix".
Container 108, Longhorn Safety and Security Department.
- 23:12 (2) Hazards - Similarity to "Soak Out" of Motors: According to data obtained in testing liquid-carrier "Quickmix" process of solid propellant manufacture, both ammonium perchlorate and mixtures of ammonium perchlorate and aluminum powder in HMDS become more hazardous as the amount of HMDS present falls below that required to fill all the voids in a settled-out mixture. The presence of aluminum enhances the hazard considerably. Hence, it is recommended that precautions be taken to guard against the possible formation of a system containing AP plus aluminum plus HMDS (hexamethyldisulfoxane). See report 23:12 (1) above.
Container 108, Longhorn Safety and Security Department.
- 23:12 (3) This report describes the features of the "Quickmix" process, the chemistry of propellants tailored for "Quickmix", and operation of Rocketdyne's 500 lb/hr pilot plant to manufacture a polyurethane solid propellant. Analysis of pilot plant data is presented, the bench scale plant is described, and recommendations for automating the pilot plant are made.
R. D. Sheeline, et al, Rocketdyne, a Division of North American Aviation.
Reference No. 8, Vol. IV, page 223.

PROPELLANTS: TRANSITION FROM DEFLAGRATION TO DETONATION

- 30:10 (1) "Measure of detonability and of the deflagration to detonation transition in solid propellants". A detailed account is given in this paper concerning the above subject. The relation of impact sensitivity to detonation and other phenomena are discussed, such as "critical diameter".
Dr. A. B. Amster, Sup. Chemist, Physical Chemistry Division, Naval Ordnance Laboratory.
Reference 6, pages 76 - 86. Thiokol Longhorn Container 108, Safety and Security Department.
- 30:10 (2) See 21:16 (2).
- 30:10 (3) Difference between "detonation" and "explosion" defined. See reference in 30:10 (1) above, page 104.
- 30:10 (4) A new approach to determination of detonability of propellants and explosives: A new quantitative approach to establishing the detonability of propellants through a study of the "Deflagration to Detonation Transition (DDT)" has shown promising results. Results thus far indicate that each explosive material may have a critical pressure above which the transition from deflagration to detonation will occur. The measurement depends on the determination of burning rate as a function of pressure. By comparing the burning rates obtained in a strand burner with those obtained for large solid cylinders in a closed bomb at high pressure, a critical pressure is found for each explosive above which the closed bomb burning rate vs pressure curve turns sharply upward from the normal burning rate vs pressure curve obtained with the strand burner.
S. Wachtell & C. E. McKnight, Picatinny Arsenal.
Reference No. 8, Vol. V, page 125.

PROPELLANTS: PHYSICAL TESTING, STATIC ELECTRICITY, IMPACT, ETC.

- 30:11 (1) A continuous oscillographic method for determining detonation velocities in solid cast explosives. Various formulations are given with test results of booster sensitivity, effect of porosity and temperature, factors causing increased port perimeter, etc.
Reference No. 3, pages 103 - 121.
- 30:11 (2) Ignition from Static Electricity: A test set-up is described to determine the ignitability of propellant with static discharge. Based on the results of experiments, it is the opinion of the writer and other persons who took part in these tests that the chance of accidental ignition from static electricity is so remote that it is practically nonexistent.
Thiokol, Huntsville Division, May 1957, "Investigation of Static Electricity with Respect to Accidental Ignition of Propellant", by J. C. Godsey, W. E. Fox, H. E. Dupree. Container 106, Longhorn Safety and Security File.
- 30:11 (3) Improvement of testing methods for determining the hazard classification of propellants: Various test equipment is described, namely, Taliani test, Impact test, Heat Cracking test, Card-Gap test, Cook-off test, and other tests. The author concludes, "The tests which are used at NOTS for the determination of the hazards associated with the handling and processing of solid propellants and propellant ingredients, are primarily empirical in nature and the interpretation of the test results in terms of practical applications is usually difficult. Because no quantitative test exists for the determination of the hazards, the key to safe operations appears to be the use of good judgment by those who determine handling and processing methods. Improved testing methods, which would minimize the need for this degree of judgment on the part of all concerned, are urgently needed".
Report by Mr. Dallett at the second meeting of the Operations Subcommittee at Roanoke, Virginia, on 2 - 3 June 1959. Topic No. 13.
Container 108, Longhorn Safety and Security File.
- 30:11 (4) Static Electricity Studies on Ether-Alcohol-Acetone: This paper covers 1) theory of static electricity, 2) equations for determining static charge accumulated on a man's body; voltage required for minimum ignition energies, and 3) static potential accumulated by airveying finished propellant. Program Officer, Office of the Technical Director.
Reference No. 6, pages 45 - 54, Container 108, Longhorn S&S Department.
- 30:11 (5) Impact Tester: "...it has been our experience that the sensitivity as measured by the falling ball does not agree with the sensitivity, or sensitivity test; that is, for material that you think is considered more hazardous, (for example, an 80% loaded oxidizer material would be considered more hazardous and more sensitive than a 60% oxidizer material of the same formulation, or the same basic degrees). However, on the falling ball impact tester, it is entirely possible that the 60% will show up as more sensitive than the 80%. I, myself, don't put an awful lot of strength in the impact sensitivity tests". Mr. Haite, Thiokol, Huntsville Division.

30:11 (5) Impact Tester: (Continued)

"I agree with you entirely that the Bureau of Mines impact test will tell you very little about what might happen in the way of a detonation. However, it is the general feeling throughout the industry that it does tell you something about the sensitivity toward ignition". Dr. A. M. Ball, Hercules Powder Company.
Reference 6, page 37.

30:11 (6) New test method for hazard evaluation for processing cast double base propellant: This paper, by Allegheny Ballistic Lab, presents their method of detecting and eliminating process hazards before an ingredient or new process is utilized in the manufacture of double base propellants. The purpose of this method is to be able to apply test data directly to the process. A "Hazard Assessment Program" is outlined. A pendulum friction machine and a sliding friction machine as used in this program are described. The hazardous properties of ammonium perchlorate are discussed. Mr. R. H. Richardson, Hercules Powder Company, Allegheny Ballistic Lab.
Reference No. 7, page 18.

30:11 (7) Gap Test for Solid Propellants: This paper outlines some of the work done at the Navy Ordnance Laboratory in investigating the basic aspects of the sensitivity of high explosives and propellants. There is no single test for sensitivity. In studying sensitivity, one must realize that the various tests employed rarely measure any basic quantity. The laboratory has accumulated a great deal of data on both liquid and solid explosives, or propellants using two "standardized" versions of the gap test. A detailed account of tests is given. Composites of inert binder type (polyurethane, polysulfide rubber, plastisol binders) and ammonium perchlorate propellants are quite insensitive as long as they are homogeneous. If they have voids in them, they become highly sensitive. Large scale gap tests (19 inches in diameter and 80 inches long "Polaris Propellant") were run by NOTS and Aerojet. No detonations were observed in 12 shots. Dr. E. C. Noonan, NOL White Oak, Silver Springs, Maryland.
Reference No. 7, pages 101 - 115.

30:11 (8) "Importance of Flexibility in Gap Sensitivity Test": The result of this investigation demonstrates that, in examining the detonability of different substances, a gap test employing variable charge dimensions offers distinct advantage over a "standard" test.
Mr. W. W. Brandon, Rohm & Haas Company.
Reference No. 8, Vol. V, Page 109.

30:11 (9) Also see 10:16, "Laboratory Incidents: Sawing, Mixing, Heating"

30:11 (10) "Static Ignition Sensitivity of Ether-Alcohol-Acetone and Propellant Dust", prepared for the Explosive Safety Seminar on High-Energy Solid Propellants, 9 - 12 June 1959, Naval Propellant Plant.

PROPELLANTS: EXPLOSIVE CLASS, TEST FOR VARIOUS PROPELLANTS, MOTORS, ETC.

- 30:12 (1) A test program was conducted at White Sands Missile Range at the request of OCO to recommend the proper explosive classification of the polysulfide-perchlorate solid propellant used in the Nike Hercules and Hawk missile systems. This paper gives data on the test run.
Mr. Frank Schultheis, White Sand Missile Range.
Reference No. 7, pages 156 - 164.
- 30:12 (2) Test procedures were established to determine an Ordnance classification for propellants used in the Minuteman propulsion system. The particular tests are described. Test results and precautions are summarized.
Lt. K. O. W. Ball, Ballistic Missile Division, Dept. of the Air Force.
Reference No. 7, pages 165 - 179.
- 30:12 (3) Hazard Classification of End Items on the Basis of Test Criteria:
This paper deals with the work and progress by the ASESB.
Mr. Herman, ASESB.
Reference No. 7, pages 152 - 155.
- 30:12 (4) See 30:13 (2), "Test to Determine Hazard Classification of Motors Larger than 12" in Diameter".
- 30:12 (5) See 30:12 (1), "Scale Testing of Motors".
- 30:12 (6) PBAA/Ammonium Perchlorate: See 12:14 (4) for hazard classification.
- 30:12 (7) Explosive Hazard Test Run on E-Series Propellant: 26 July 1961. Thiokol, Huntsville Division, Memorandum from F. S. Monteleone, to W. F. Haite, Manager, Propellant and Process Development Program.

PROPELLANTS: SCALE TESTING OF MOTORS

- 30:13 (1) Scale Testing of Motors: Mr. R. C. Herman, ASESB, "...This problem of hazard classification has caused considerable difficulty. The Board has attempted to develop a standard test criteria...so we came out with a minimum test criteria for various items. We put in a statement that, until information was developed, we would have to use full scale items in testing these large motors. Everyone said they couldn't do it, it would cost too much money. A meeting was held on the 28th of May 1959, but we finally came to an agreement of using scale model items and the criteria for this was based on what Dr. Amster gave us--this equivalent diameter, with a length of about $3\frac{1}{2}$ times the diameter. At present, the criteria calls for utilization of full size motors up to 17 inches. Greater than this, you will use this scale model which is a grain diameter equal to the equivalent diameter and a length of $3\frac{1}{2}$ times this". Reference 6, page 100.
- 30:13 (2) Problems in establishing explosive hazard classifications of solid propellant rocket motors larger than 17 inches in diameter, is discussed. First, what are the minimum test criteria for determining the hazard classification of large propellant rocket motors? Second, will data obtained from sub-scale motors be in order to provide valid data? The Department of Defense publishes a bulletin which outlined the minimum test criteria to assure uniform assignment of hazard classification to a given item by all agencies in each of the services. This bulletin is a coordination publication. See Department of the Army Technical Bulletin 700-2, Department of the Navy NAVORD Instruction 8020.8, or the Department of the Air Force Technical Order 11A-1-47. On the basis of these considerations, the 2705th Air-Munitions Wing at Ogden Air Material Area has undertaken a project to determine the minimum test criteria for solid propellant rocket motors larger than 17 inches in diameter and to determine if it will be possible to use scaled motors. Mr. S. H. Welch, OQAMA. Reference No. 7, page 147.
- 30:13 (3) Using scale rocket motor performance as a basis for the acceptance of large Sergeant rocket motors. Jet Propulsion Laboratory, Sperry, Utah Engineering Laboratory. Reference No. 8, Vol. V, page 147.

PROPELLANTS: PHYSICAL STATE, POROSITY, GRAIN SIZE

- 30:14 (1) In explosive mixtures which exhibit connected porosity, the deflagration-to-detonation mechanism appears to be relatively easy to establish, so that all finely divided solid propellants tested to date exhibit DTD when ignited under sufficient confinement.
Reference No. 3, page 123.
- 30:14 (2) Introduction of even a small amount of porosity in composite and double base propellants increases the sensitivity markedly, in some cases rendering the propellant more sensitive than tetryl.
Reference No. 3, page 89.

PROPELLANT: EFFECTS OF HIGH-ENERGY RADIATION

- 30:15 (1) Solid propellants and fuels were cured quickly by Gamma radiation, without heat, to open a new approach to propellant manufacture. The optimum time of cure, corresponding to total doses of about 10^6 rep, were small fractions of the normal thermal cycle. Cure of polyurethane propellants at ambient temperature has been unsuccessful thus far.
Aerojet-General Corporation.
Reference No. 3, pages 137 - 200.

SAFETY DESIGN CRITERIA: MANUFACTURING AND STORAGE BUILDINGS

- 40:10 (1) "Establishment of Safety Design Criteria for High-Energy Propellant Manufacturing and Storage Facilities", Picatinny Arsenal. Test methods employed for determining sensitivity, impact, heat, and detonability (test in heavy wall pipe by means of a booster, etc.) of high-energy propellants are of value up to a point, but they do not give a complete quantitative picture (see Picatinny Arsenal Technical Report DB-Tr: 13-58, H. L. Partridge, Safety Hazards of Rocket Propellants, November 1958). The designer must take into consideration such factors as propellant output, a sensitivity to blast, and sensitivity to fragment impact. In line with the above, Picatinny Safety Design Criteria Program has been divided into three major phases: Phase I deals with establishment of realistic quantity-distance relationships for prevention of sympathetic detonation; Phase II, the effects of fragment impact in causing high order detonation in an explosive charge, and related safety design criteria; Phase III relates to structural design criteria for barricades, substantial dividing walls, and other protective structures.
Reference No. 6, pages 109 - 181.
- 40:10 (2) "Determination of propellants and safety design criteria for manufacture and storage", Picatinny Arsenal. A review of the above (40:10 - 1) is given. Mathematical formulas, drawings, etc., are given, as: striking velocity of a fragment as a function of fragment mass and distance.
Mr. L. W. Scaffian, Picatinny Arsenal.
Reference No. 7, pages 53 - 60. Container 108, Longhorn S&S Dept. File.

SAFETY DESIGN CRITERIA: PROPELLANT EXTINGUISHING SYSTEMS

- 40:11 (1) A water quench system for individually loaded motors in ship magazines that is automatically activated within $\frac{1}{4}$ to $\frac{1}{2}$ second after ignition of the motor, is discussed.
Mr. Floyd, Applied Physics Laboratory, John Hopkins University.
Reference No. 6, pages 173 - 175.
- 40:11 (2) Fire detection and control for solid propellants: A single compact unit consisting of a detector, valve, nozzle, and water under pressure, is used to detect and extinguish a motor if accidentally ignited. Application in cut-back operation is discussed.
Mr. C. F. Averill, Grinnell Corporation.
Reference No. 7, page 95.
- 40:11 (3) This paper is a review of the merits of water injection systems. Various problems are reviewed. Experiments with different motor fires are given. Water pressure, quantity, etc., are shown.
Mr. S. H. McElroy, Navy Weapons Laboratory, Dahlgren, Virginia.
Reference No. 7, page 130.
- 40:11 (4) Tests of Production Terrier Booster Quench Nozzles manufactured by the Automatic Sprinkler Corporation. U. S. Naval Weapons Laboratory, Dahlgren, Virginia, 13 May 1960, Report 1703.
Thiokol Longhorn Container 108, Safety and Security Department File.
- 40:11 (5) Mixer Fire Detection and Extinguishing Device: A 100-gal' water tank located above the mixer is equipped with an infra-red cell. This cell, on detecting a sudden temperature rise, ignites a propellant charge which pressurizes the water tank. Below the tank is attached a 6-inch pipe which projects inside the propellant vessel. The end of the pipe is closed with a thin aluminum disk, and as soon as the water vessel is pressurized (above about 100 p.s.i.), this disk bursts, allowing a 6-inch jet of water to enter the mix. This automatic drencher system has proved most effective in quenching simulated fires.
Ministry of Aviation, Explosive Research and Development Establishment, Waltham Abbey, Essex, United Kingdom.
Reference No. 8, Vol. V, page 54.
- 40:11 (6) Mixer Fire Detection and Extinguishing Device: Rocketdyme, McGregor, Texas. Ammonium nitrate propellant fire in a 100-gallon Baker Perkins mixer extinguished with only minor damage. Operation of vacuum-stat raised mixer lid and propellant fire extinguished by water deluge, 22 June 1962. Telegram, RT-1188 - Thiokol Longhorn Safety File 1/304.4.6.
- 40:11 (7) Research on Chemical Extinguishment of Solid Propellant, Thiokol Chemical Corporation, Elkton Division. Elkton Project No. 7698. TCC-Longhorn. Technical Library No. 2307. (The concept of using Freon 1301 as the basis of a propellant extinguishing system that would operate with only a small weight of extinguishing agent does not look promising.)

SAFETY DESIGN CRITERIA: BARRICADES, PANELS, SHIELDS

- 40:12 (1) Comparative Test of Barricade Material: Observation windows and blast mats were exposed to test explosions. Plexiglas was found superior to bullet-proof glass for windows. Also, covering the edges of the windows markedly increases strength. Test results with 1 to 20 lbs. of C-4 explosive packed in steel pipe. Ability of Plexiglas, plate glass, blast mats, steel plate, and sandbags to withstand the explosions is shown. Dr. Knapp, Esso Research and Engineering Company. Reference No. 7, page 32.
- 40:12 (2) A Laboratory Shield for Protection Against an Explosion or Fire: The shields are of the sliding door type and made of safety glass. Dr. S. C. Burket, Aerojet-General Corporation. Reference No. 7, page 13.
- 40:12 (3) Pressure and Blast Relief Panel Test: Test conducted by Thiokol at Longhorn. Test indicated that translucent plastic is better for relieving pressure than cement asbestos, in that it relieves at lower pressure without fragmenting. Safety and Security Department File, Feb. 27-28 and March 1-2, 1961.
- 40:12 (4) Safety in Handling High-Energy Propellant Ingredients in the Laboratory: By C. L. Knapp, Esso Research and Engineering Company. Gives new design of equipment, as compact robot manipulator, laboratory layout, etc. Reference Bulletin of the 17th Meeting of JANAF Solid Propellant Group, Vol. I, page 143.

SAFETY REQUIREMENTS: MOTOR TESTING, CONDITIONING

- 50:10 (1) "Firing Test Cell Design and High Temperature Testing." Technique used to safely test solid propellants in the range of 300° to 500°F, is described. Safety precautions from a standpoint of test cell design and handling is discussed.
Dr. Burket, Aerojet-General.
Reference No. 7, pages 126 - 129.
- 50:10 (2) Description of some of the instrumentation techniques used in a gas phase investigation of the "deflagration-to-detonation transition" phase of propellant combustion.
17th Meeting, JANAF Solid Propellant Static Test Panel.
- 50:10 (3) "Prediction of Catastrophic Motor Explosion Conditions from Broad Spectrum Mechanical Property Analysis." E. I. du Pont De Nemours and Company.
Reference No. 8, Vol. V, page 61.
- 50:10 (4) AOMC has completed the first in a series of auto-ignition studies on the XM30 motor. A motor, less pyrogen and adapter, was subjected to an increasing temperature environment from ambient to approximately 325°F. Ignition of the motor was accomplished at 325°F as indicated by thermocouples manually inserted in the propellant grain. Future tests will be performed, utilizing the missile system heating blanket as a heat source.
Reference, Weekly Management Bulletin, 2 April 1962, Thiokol, Longhorn.
- 50:10 (5) AOMC has completed an auto-ignition test on the XM30 motor using the tactical heating blankets. At a power setting to the blankets, a motor case skin temperature of 265°F was maintained for approximately 8 days. Following this time, the power to the blankets was increased to the point the motor case skin temperature increased to 310°F, which correlated to a maximum grain temperature reading of 233°F. The motor ignited following 18 hours exposure time to this condition.
Reference, Weekly Management Bulletin, 11 June 1962, Thiokol, Longhorn.

SAFETY REQUIREMENTS: RADIO FREQUENCY ENERGY

- 50:11 (1) "Radio Frequency as Related to Accidental Ignition of Electrical Squibs by Induced Current." This paper deals with three particular problems that the JPL had in this area -- the Sergeant Missile, the RTV vehicle, and the on-lab R&D operations at the laboratory--and the approach which we took to solve these problems. The two squibs studies are Dupont X201A and Dupont X201E. Radio frequency energy can be supplied to a squib from a firing harness which acts as an antenna. The problems, incidents, and methods to prevent accidental ignition are discussed.
Mr. L. R. Piasecki, JPL.
Reference No. 7, page 116.

SAFETY REQUIREMENTS: BURNING GROUNDS, SCRAP COLLECTION, VACUUM SYSTEMS

50:12 (1) See 11:21

50:12 (2) Discussion of scrap collection, safety shoes, vacuum collection of cutback fires, burning ground problems.
Reference No. 6, pages 86 - 96.

50:12 (3) See 11:14 (4) - Control of atmospheric concentrations of aluminum powder by use of a wet-type dust collector.

50:12 (4) See 10:15 (6) - Vacuum Line Explosion.

50:12 (5) Free Flight of Casting Hose: Thiokol, Longhorn Division, 1956.
When burning scrap propellant containing a section of propellant-filled casting hose, the hose free-flighted vertically 300 - 400 feet. Casting hose are now burned in a cage.